



1992

## PERFORMANCE REPORT

Drinking Water Organics Section

Eva Duchoslav (ed.)  
Drinking Water Analyses Section  
Laboratory Services Branch  
Ministry of Environment and Energy

June 1, 1993

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Performance Summary, 1992  
Drinking Water Organics Section

MINISTRY OF THE ENVIRONMENT

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### **ACKNOWLEDGEMENT**

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*Drinking Water Organics Section*

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## **INTRODUCTION**

This document provides the 1992 summary of the performance of routine approved analytical methods within the laboratories of the former Drinking Water Organics Section ( DWO ), namely the Dioxin Unit, the Mass Spectrometry Unit, the Organic Water Unit and the Priority Pollutants Unit.

In December 1992 the Laboratory Services Branch was reorganized. The Plasma Spectrometry/ Trace Metals Unit was added to the Drinking Water Organics Section and to form the Drinking Water Analyses Section ( DWA ).

The Drinking Water Analyses Section is responsible for both qualitative and quantitative analyses of drinking, surface, river and lake waters for trace metals, major elemental components and a wide array of organic chemicals, such as chlorinated benzenes, herbicides, pesticides, polynuclear aromatic hydrocarbons, extractable organics and purgeable organics at the part-per-trillion or part-per-billion level. In addition, analyses provided by the Section include ultra-trace quantitative analyses of all environmental matrices for polychlorinated dibenzodioxins and polychlorinated dibenzofurans, most notably 2,3,7,8-tetrachlorodibenzo-p-dioxin.

The major objective of the DWA Section's quality assurance program is to produce data of known quality, appropriate to the particular project requirements. The quality of the data is to be supported by documentation acceptable to scientific community, and gathered in accordance with the established ministry protocols. The quality control program is designed to detect any anomalies in the quality of the analytical results and to provide the basis for an immediate corrective action.

Within the DWA Section, the most common quality control tasks include the analyses of quality control samples, such as method blanks, fortified method blanks, samples fortified with surrogates, check calibration solutions and reference materials, and the interpretation of the resulting data. For each analytical method, the actual quality control procedures are described in detail in the official method text.

This Performance Summary Report is based on the results of selected quality control samples acquired in the Drinking Water Organics Section between January and December, 1992. In this report, each abstract of the analytical method is accompanied by corresponding performance charts and summary tables. Performance charts indicate the mean and the 99%-confidence limits for the variable presented.

The Plasma Spectrometry / Trace Metals Unit was incorporated into the DWA at the end of 1992 and therefore, data from this Unit is not reported here.

## GLOSSARY OF TERMS

|                            |   |
|----------------------------|---|
| accuracy                   | proximity to the true value expressed as average percent recovery or average percent of expected  |
| average (mean)             | sum of the measurements divided by the number of measurements   |
| between-run experiment     | samples are prepared by different technicians, and the instrumental analyses take place under different calibrations of the analytical system   |
| between-run r.s.d.         | measure of reproducibility of a method  |
| calibration solution       | a solution containing target analyte(s) for a particular method at concentration(s) that will produce response(s) falling within the linear range of the instrument. This solution is used to calibrate the instrument response with respect to the analyte concentration.      |
| calibration check solution | a solution of a composition similar to the calibration solution, prepared independently of the calibration solution. It is used to check performance of the instrument, especially, the validity of current calibration.  |
| fortified method blank     | a synthetic sample prepared by adding known quantities of the analytes of interest to the interference-free matrix  |
| IDL                        | instrumental detection limit. The concentration giving an instrumental response of 5:1 signal-to-noise height ratio.  |
| internal standard          | a known amount of a compound, that is assumed to have identical chemical and physical properties with the analyte(s) of interest, is added to the sample prior to sample processing. The recovery of this compound from the sample is used for correction of the final results. |
| MDL                        | method detection limit. MDL marks the concentration level above which one can conclude that a measured result indicates the presence of analyte in the sample with a specified confidence (99%).  |
| method code                | Analytical Methods Catalogue Code used within Ontario Ministry of the Environment   |
| percent recovery           | ratio of the concentration obtained by the experiment to the theoretical concentration, multiplied by one hundred   |

performance charts

graphical presentation of the individual results of the analyses of fortified method blanks. The x-axis on the chart represents the date, the y-axis outlines percent recovery. The average and 99% confidence limits are displayed as well.

standard deviation

measure of spread of a population. The square root of the squared sum of the measurements minus the sum of squared measurements, divided by the number of measurements minus one.

T value

level below which analytical results represent trace values; additional data are needed for valid interpretation ( see Code of Practice for Environmental Laboratories, September 1989, Ontario Ministry of the Environment )

upper and lower 99% confidence limit, UL (LL)

$UL (LL) = X + (-) t \times s$   
X, s represent the average and the standard deviation of the replicate measurement;  
 $t_{(n-1,\alpha=0.01)}$  is the Student's t-value appropriate for a 99% confidence level and the given number of degrees of freedom n

within-run experiment

samples are prepared and analyzed by a single technician, and the instrumental analyses take place within one calibration of the analytical system

within-run r. s. d.

measure of repeatability of a method

W value

minimum reported level ( see Code of Practice for Environmental Laboratories, September 1989, Ontario Ministry of the Environment )

**METHOD CODE :** OPOV-E3144B  
**METHOD TITLE:** The Determination of Volatile Organic Compounds in Raw and Treated Drinking Water by Dual Capillary Column Dual FID / Purge and Trap Gas Chromatography  
**LABORATORY :** Priority Pollutants Unit  
**SUPERVISOR :** Dr. W. Berg  
**SAMPLE TYPE :** raw and treated drinking water, surface water, groundwater

**PRINCIPLE OF THE METHOD :**

Volatile organic compounds in water are determined by purge-and-trap technique, followed by dual capillary column gas chromatography with dual flame ionization detection. The volatile organic compounds are purged from the sample with helium at room temperature onto an adsorbent trap. The compounds are then thermally desorbed and, prior to being introduced to the gas chromatograph, are focused cryogenically with liquid nitrogen. Target compounds are quantified by an external standard calibration method.

| PARAMETERS MEASURED :    | LIS TEST CODE | W ( µg/L ) | T ( µg/L ) |
|--------------------------|---------------|------------|------------|
| v vinyl chloride         | X1022P        | 0.05       | 0.5        |
| 1,1-dichloroethene       | X1001P        | 0.05       | 0.5        |
| dichloromethane          | X1002P        | 0.5        | 5.0        |
| trans-1,2-dichloroethene | X1003P        | 0.05       | 0.5        |
| 1,1-dichloroethane       | X1004P        | 0.05       | 0.5        |
| cis-1,2-dichloroethene   | X1CDCE        | 0.05       | 0.5        |
| chloroform               | X1005P        | 0.1        | 1.0        |
| 1,1,1-trichloroethane    | X1006P        | 0.05       | 0.5        |
| 1,2-dichloroethane       | X1007P        | 0.1        | 1.0        |
| carbon tetrachloride     | X1008P        | 0.2        | 2.0        |
| benzene                  | B2001P        | 0.05       | 0.5        |
| 1,2-dichloropropane      | X1009P        | 0.05       | 0.5        |
| trichloroethylene        | X1010P        | 0.05       | 0.5        |
| bromodichloromethane     | X1011P        | 0.2        | 2.0        |
| toluene                  | B2002P        | 0.05       | 0.5        |
| 1,2-dibromoethane        | X2EDB         | 0.1        | 1.0        |
| 1,1,2-trichloroethane    | X1012P        | 0.1        | 1.0        |
| dibromochloromethane     | X1013P        | 0.2        | 2.0        |
| tetrachloroethylene      | X1014P        | 0.05       | 0.5        |
| chlorobenzene            | X2001P        | 0.05       | 0.5        |
| ethylbenzene             | B2003P        | 0.05       | 0.5        |
| m-xylene                 | B2005P        | 0.05       | 0.5        |
| p-xylene                 | B2004P        | 0.05       | 0.5        |
| bromoform                | X1015P        | 0.5        | 5.0        |

(parameters measured continued)

|                           |        |      |     |
|---------------------------|--------|------|-----|
| styrene                   | B2008P | 0.05 | 0.5 |
| o-xylene                  | B2006P | 0.05 | 0.5 |
| 1,1,2,2-tetrachloroethane | X1016  | 0.1  | 1.0 |
| 1,4-dichlorobenzene       | X2002P | 0.05 | 0.5 |
| 1,3-dichlorobenzene       | X2003P | 0.05 | 0.5 |
| 1,2-dichlorobenzene       | X2004P | 0.05 | 0.5 |
| total trihalomethanes     | X2TTHM | 0.5  | 5.0 |

**REPORTING FORMAT :**

Results are reported in parts per billion ( $\mu\text{g/L}$ ) rounded off to the closest increment of W and up to maximum of three significant figures.

**QUALITY CONTROL :**

The routine quality control operations monitor absence of potential interferences (method blanks) and consistency with the predetermined method performance (fortified method blanks). The control limits for the percent recovery of the target analytes from the fortified method blanks are set at 85% and 115%.

**REMARKS :** In addition to the intra-laboratory method control, the performance of the method was examined through performance audit samples program organized by LSB QM Office.

In 1992, the method participated in the VCM+BTX Roundrobin Study ( E690 ) organized by Esso Chemical Canada and in the Smithville Tender Intercomparison Study organized by the Laboratory Services Branch.

List of Performance Charts : all analytes, except vinyl chloride and cis-1,2-dichloroethene, described as parameters measured ( recoveries from fortified blanks )

List of Performance Tables : Method Blanks Summary  
all analytes, except vinyl chloride and cis-1,2-dichloroethene, described as parameters measured

Method Blanks Summary

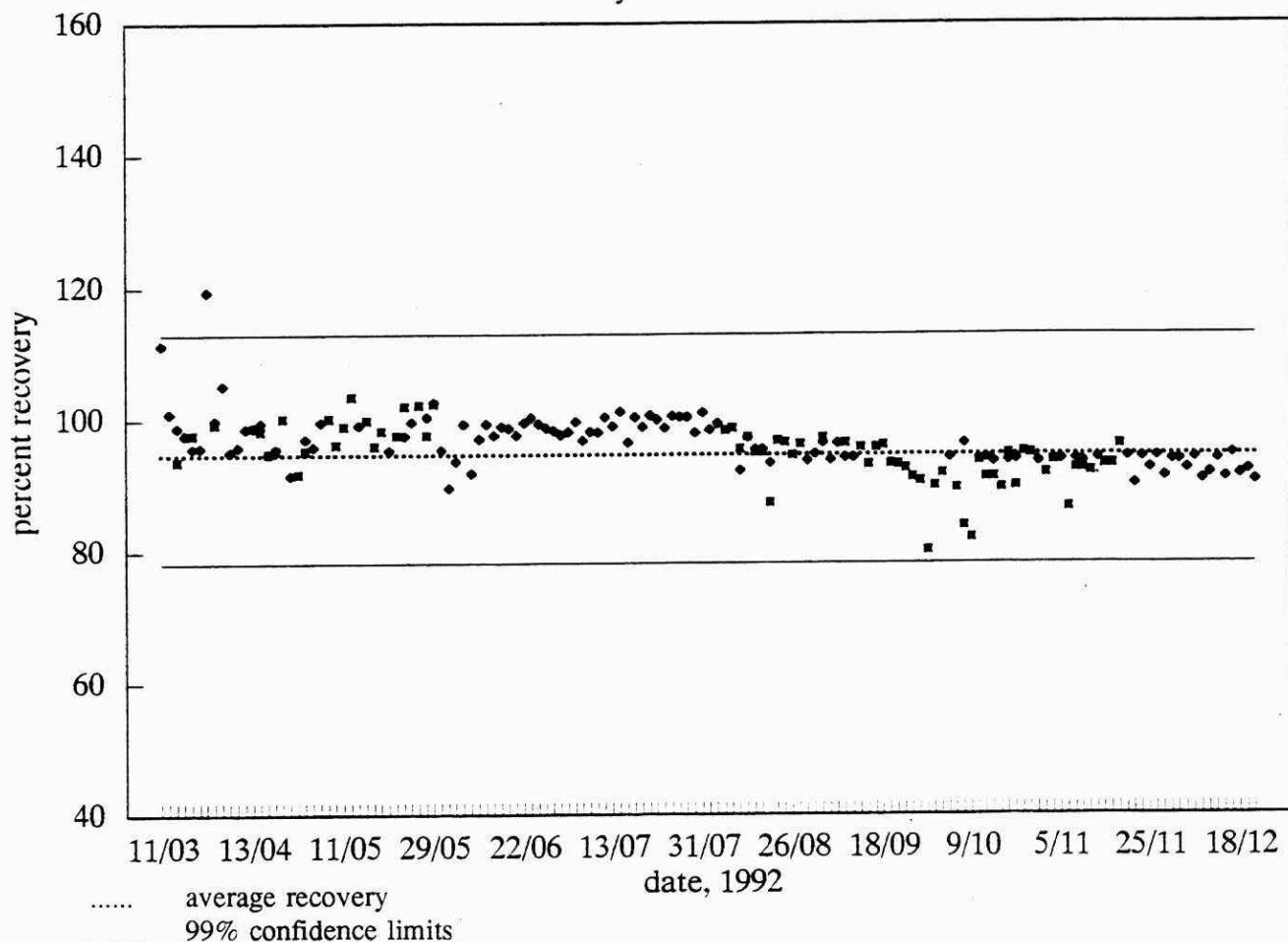
January 1992 - December 1992

| Analyte                   | Number of Observations | Average Concentration ( µg/L ) | Standard Deviation ( µg/L ) |
|---------------------------|------------------------|--------------------------------|-----------------------------|
| vinyl chloride            | 363                    | ND ( 0.02 )                    |                             |
| 1,1-dichloroethene        | 363                    | 0.0002                         | 0.0023                      |
| dichloromethane           | 363                    | 0.44                           | 0.52                        |
| trans-1,2-dichloroethene  | 363                    | ND ( 0.02 )                    |                             |
| 1,1-dichloroethane        | 363                    | ND ( 0.02 )                    |                             |
| cis-1,2-dichloroethene    | 363                    | ND ( 0.02 )                    |                             |
| chloroform                | 363                    | 0.01                           | 0.29                        |
| 1,1,1-trichloroethane     | 363                    | 0.0002                         | 0.0024                      |
| 1,2-dichloroethane        | 363                    | 0.00003                        | 0.00043                     |
| carbon tetrachloride      | 363                    | ND ( 0.01 )                    |                             |
| benzene                   | 363                    | 0.0048                         | 0.0062                      |
| 1,2-dichloropropane       | 363                    | ND ( 0.02 )                    |                             |
| trichloroethene           | 363                    | 0.0004                         | 0.0038                      |
| bromodichloromethane      | 363                    | 0.002                          | 0.022                       |
| toluene                   | 363                    | 0.010                          | 0.020                       |
| 1,2-dibromoethane         | 363                    | ND ( 0.1 )                     |                             |
| 1,1,2-trichloroethane     | 363                    | ND ( 0.05 )                    |                             |
| dibromochloromethane      | 363                    | ND ( 0.2 )                     |                             |
| tetrachloroethene         | 363                    | ND ( 0.04 )                    |                             |
| chlorobenzene             | 363                    | 0.00004                        | 0.00055                     |
| ethylbenzene              | 363                    | 0.0006                         | 0.0023                      |
| m-xylene / p-xylene       | 363                    | 0.005                          | 0.019                       |
| bromoform                 | 363                    | 0.002                          | 0.0029                      |
| styrene                   | 363                    | 0.0003                         | 0.0021                      |
| o-xylene                  | 363                    | 0.0011                         | 0.0044                      |
| 1,1,2,2-tetrachloroethane | 363                    | 0.011                          | 0.072                       |
| 1,4-dichlorobenzene       | 363                    | 0.0022                         | 0.0082                      |
| 1,3-dichlorobenzene       | 363                    | 0.003                          | 0.012                       |
| 1,2-dichlorobenzene       | 363                    | 0.0023                         | 0.009                       |

ND ... Not detected. Detection limit in µg/L given in brackets ( ).

## 1,1-dichloroethene

recovery from fortified blank



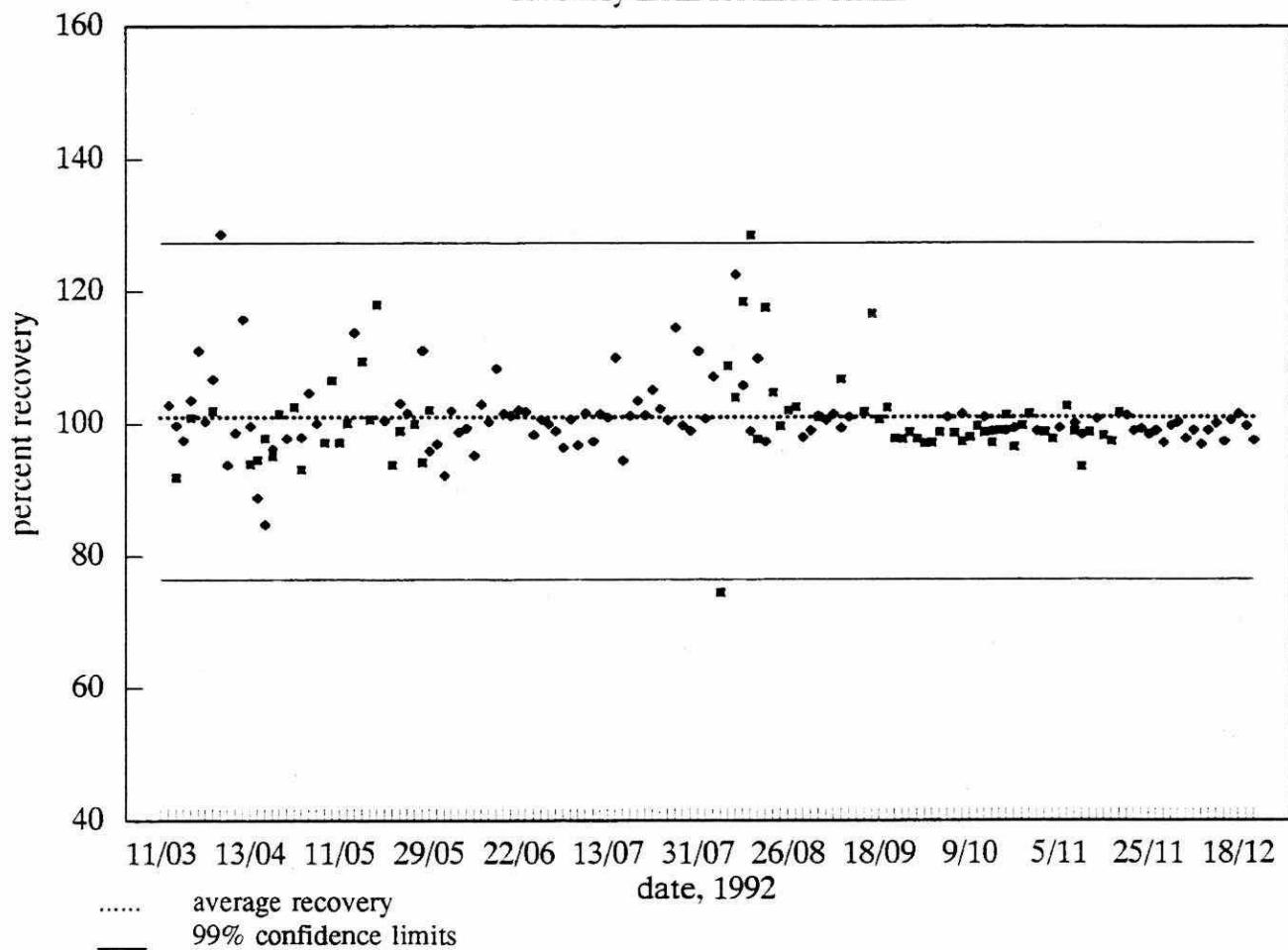
Performance Summary Table

January - December 1992

| Analyte                            | 1,1-dichloroethene |
|------------------------------------|--------------------|
| True Concentration                 | 3.68 µg/L          |
| Number of Observations             | 172                |
| Within-run Rel. Standard Deviation | 0.8% (n=7)         |
| Between-run Standard Deviation     | 6.8%               |
| Accuracy (% of expected)           | 95.6%              |

## dichloromethane

recovery from fortified blank



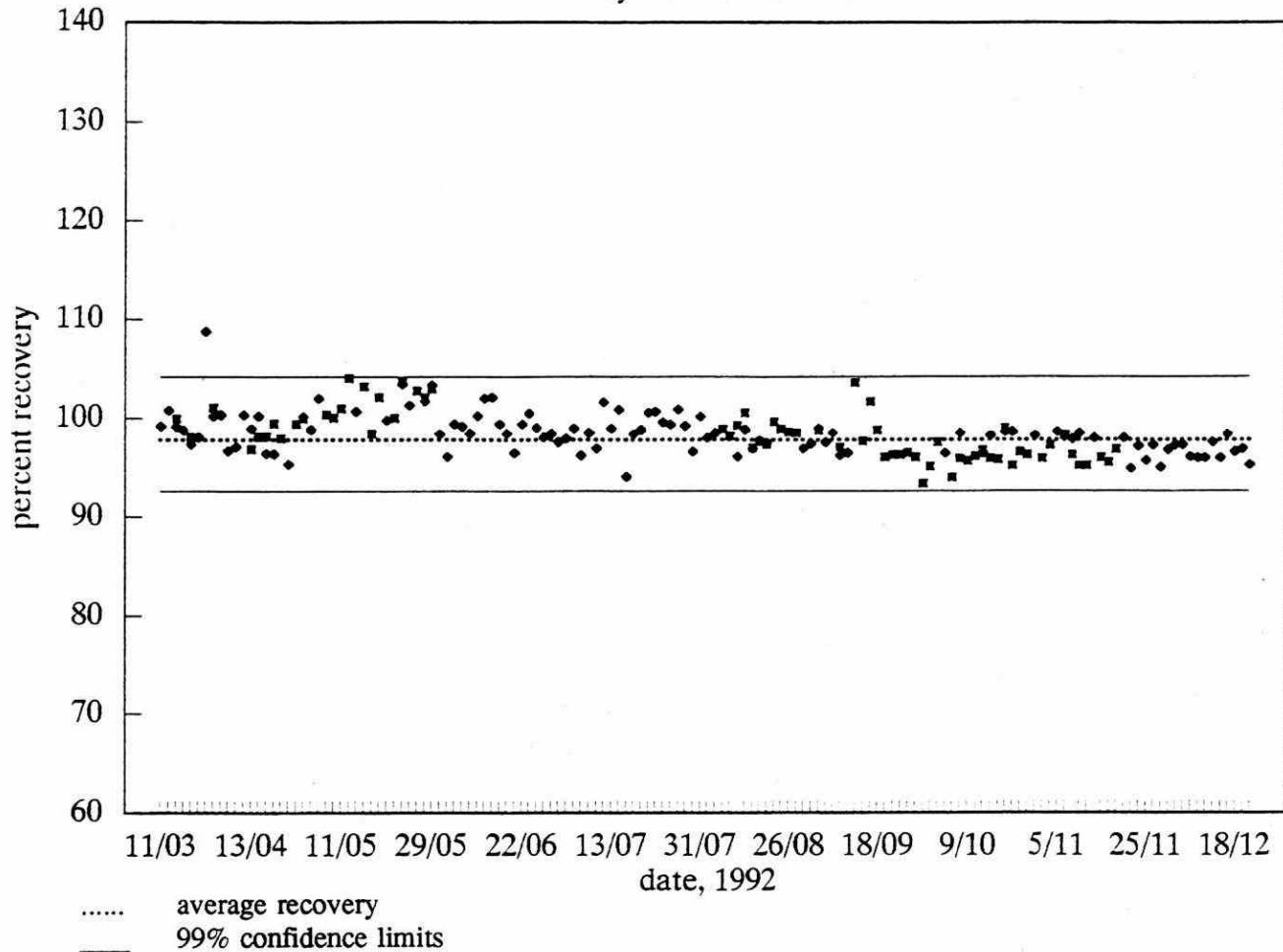
Performance Summary Table

January - December 1992

| Analyte                            | dichloromethane |
|------------------------------------|-----------------|
| True Concentration                 | 3.68 µg/L       |
| Number of Observations             | 172             |
| Within-run Rel. Standard Deviation | 2.2% (n=7)      |
| Between-run Standard Deviation     | 10%             |
| Accuracy (% of expected)           | 102%            |

## t-1,2-dichloroethene

recovery from fortified blank



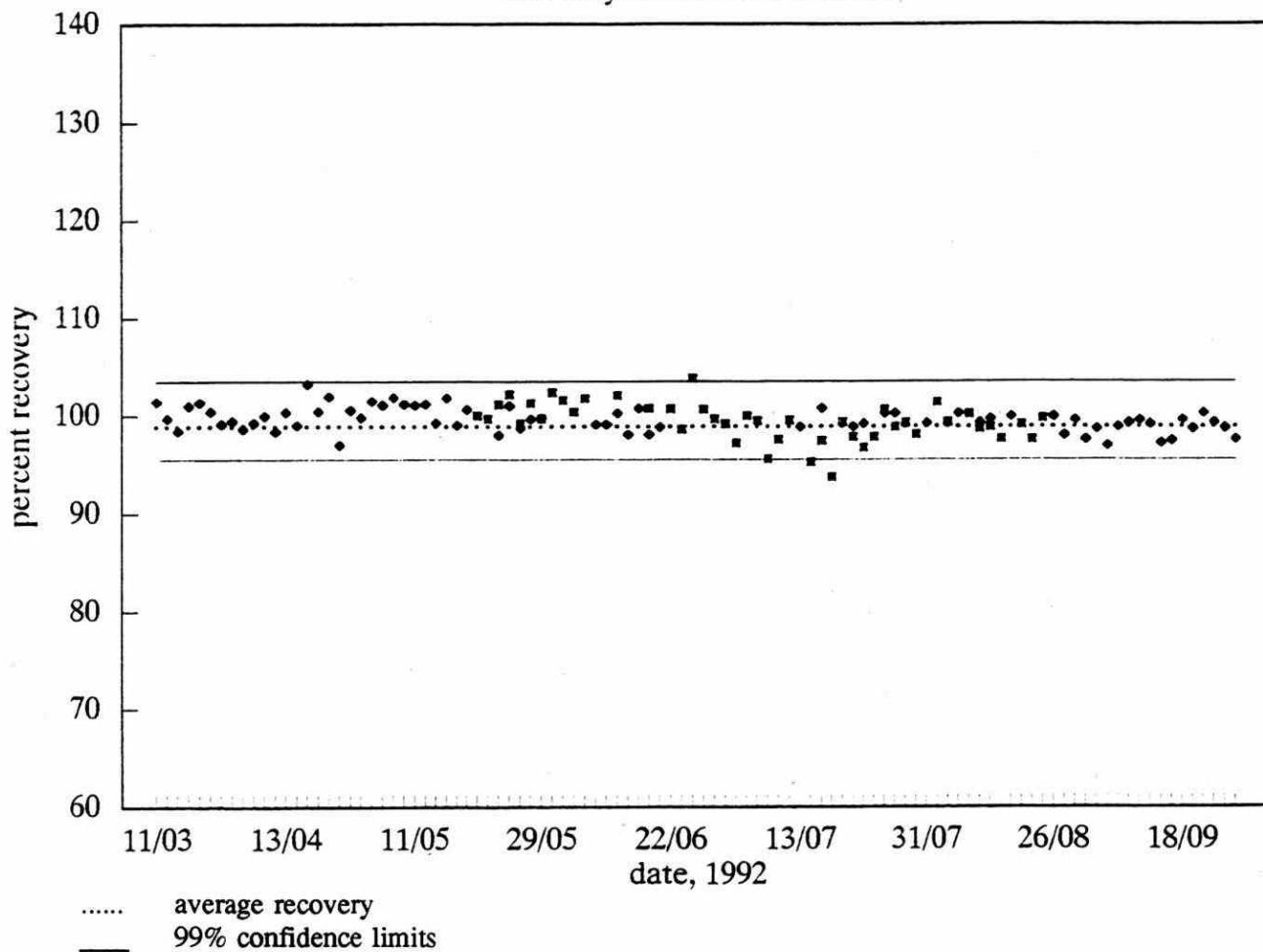
Performance Summary Table

January - December 1992

|                                    |                          |
|------------------------------------|--------------------------|
| Analyte                            | trans-1,2-dichloroethene |
| True Concentration                 | 3.68 µg/L                |
| Number of Observations             | 172                      |
| Within-run Rel. Standard Deviation | 1.1% (n=7)               |
| Between-run Standard Deviation     | 2.3%                     |
| Accuracy (% of expected)           | 98.4%                    |

## 1,1-dichloroethane

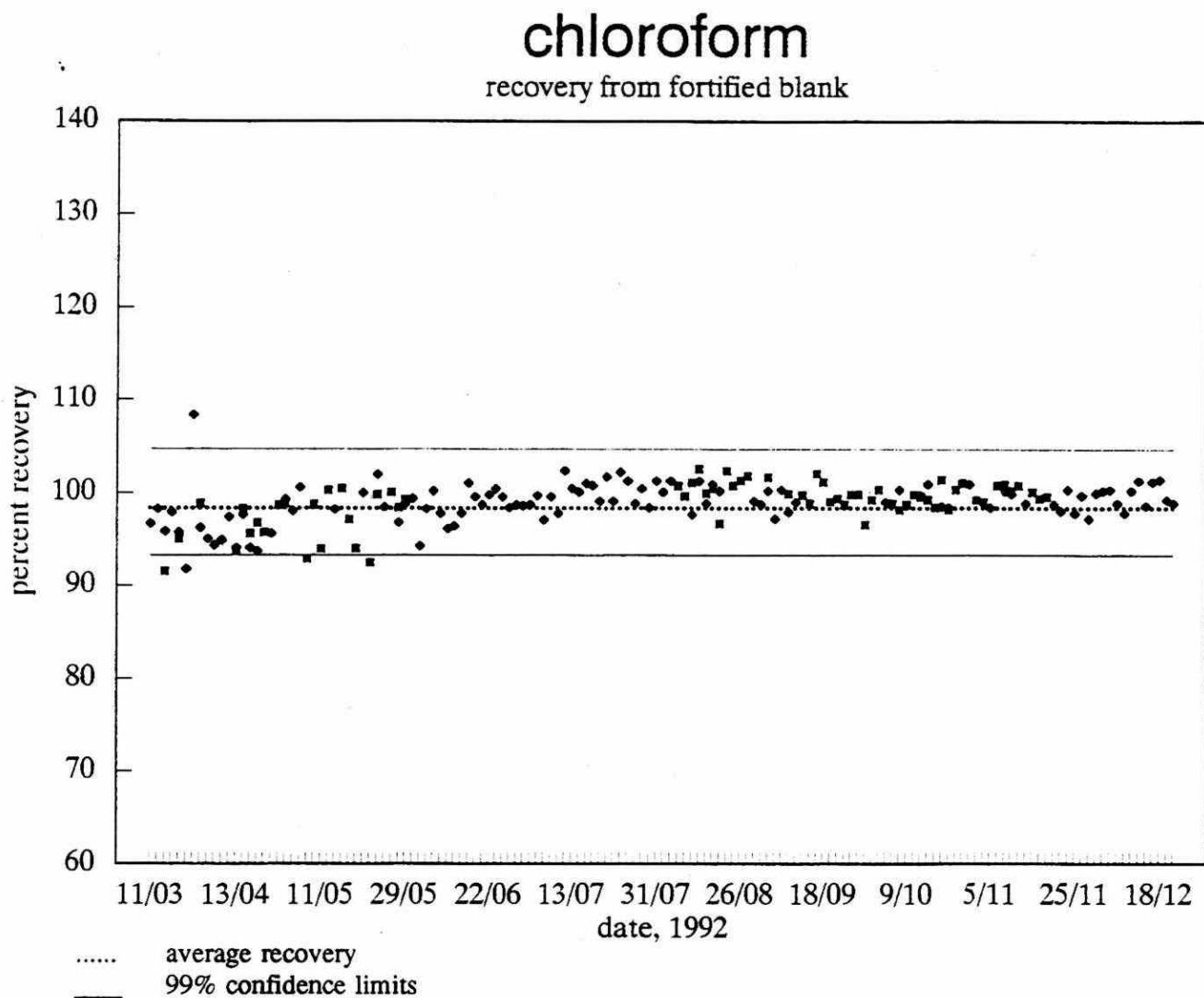
recovery from fortified blank



Performance Summary Table

January - December 1992

|                                    |                    |
|------------------------------------|--------------------|
| Analyte                            | 1,1-dichloroethane |
| True Concentration                 | 3.68 µg/L          |
| Number of Observations             | 116                |
| Within-run Rel. Standard Deviation | 1.1% (n=7)         |
| Between-run Standard Deviation     | 1.6%               |
| Accuracy (% of expected)           | 99.5%              |



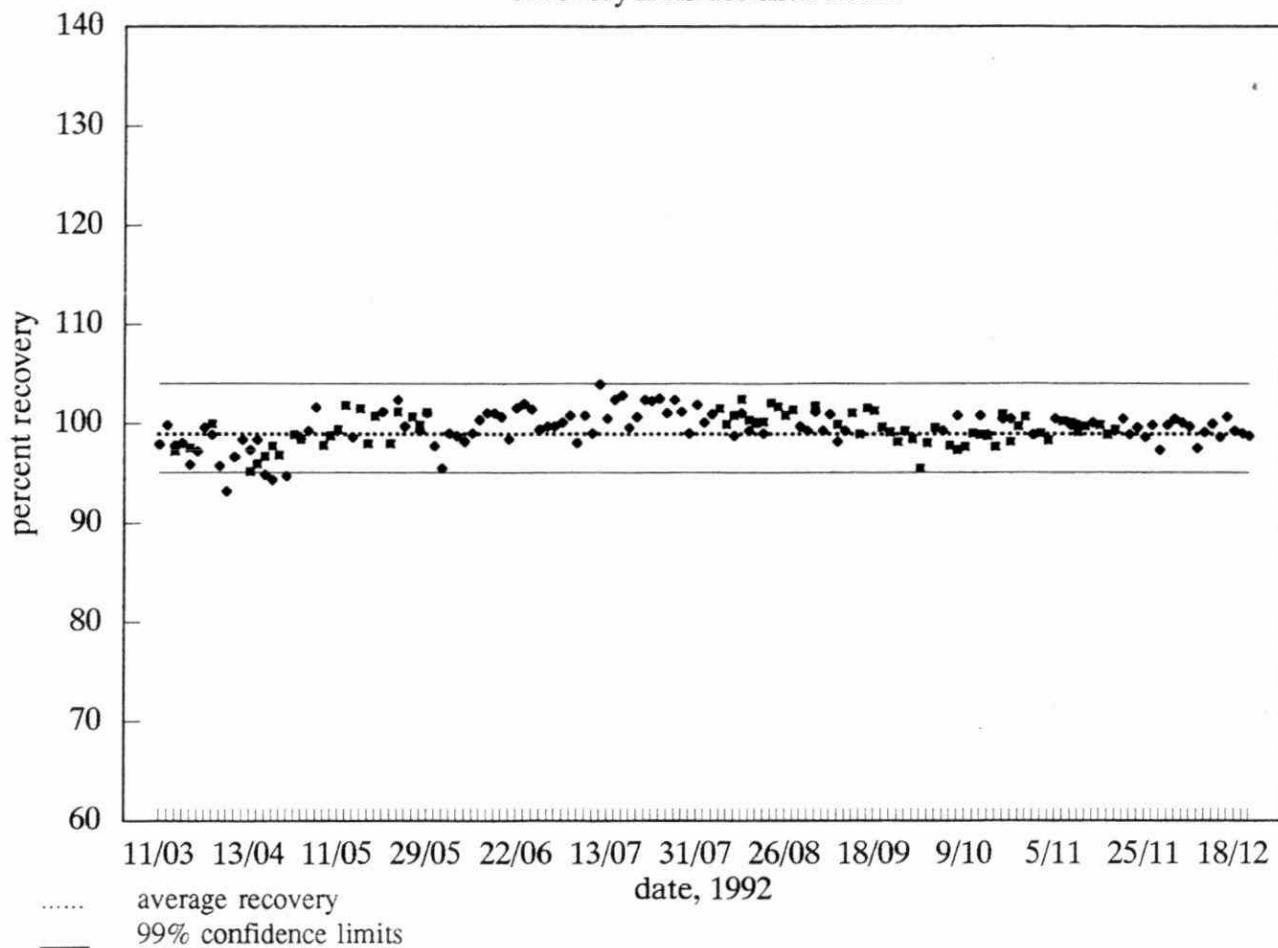
Performance Summary Table

January - December 1992

|                                    |            |
|------------------------------------|------------|
| Analyte                            | chloroform |
| True Concentration                 | 3.68 µg/L  |
| Number of Observations             | 172        |
| Within-run Rel. Standard Deviation | 2.5% (n=7) |
| Between-run Standard Deviation     | 2.2%       |
| Accuracy (% of expected)           | 99.0%      |

## 1,1,1-trichloroethane

recovery from fortified blank



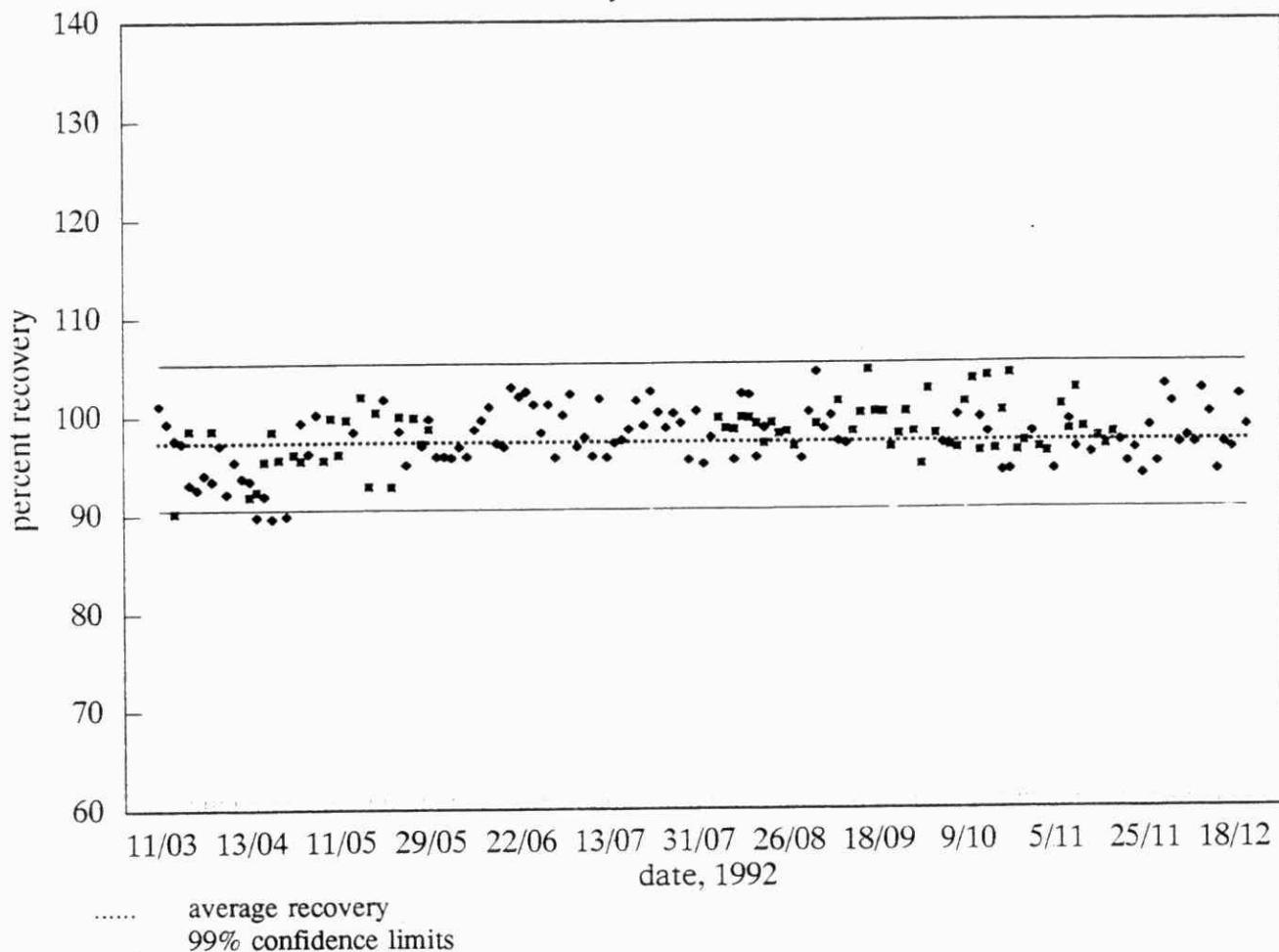
Performance Summary Table

January - December 1992

|                                    |                       |
|------------------------------------|-----------------------|
| Analyte                            | 1,1,1-trichloroethane |
| True Concentration                 | 3.68 µg/L             |
| Number of Observations             | 172                   |
| Within-run Rel. Standard Deviation | 1.1% (n=7)            |
| Between-run Standard Deviation     | 1.8%                  |
| Accuracy (% of expected)           | 99.5%                 |

## tetrachloromethane

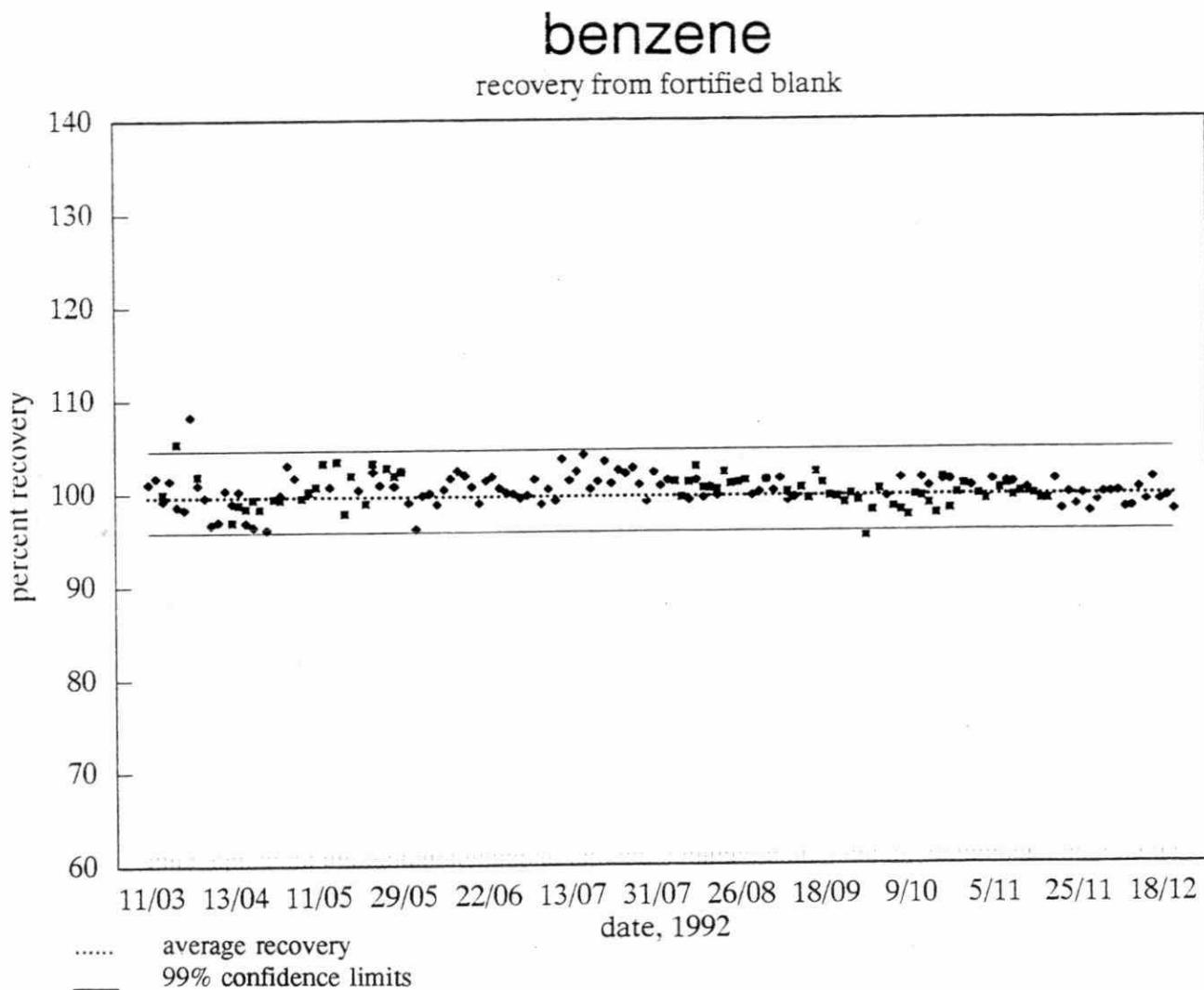
recovery from fortified blank



Performance Summary Table

January - December 1992

| Analyte                            | tetrachloromethane |
|------------------------------------|--------------------|
| True Concentration                 | 3.68 µg/L          |
| Number of Observations             | 172                |
| Within-run Rel. Standard Deviation | 1.8% (n=7)         |
| Between-run Standard Deviation     | 2.9%               |
| Accuracy (% of expected)           | 97.9%              |



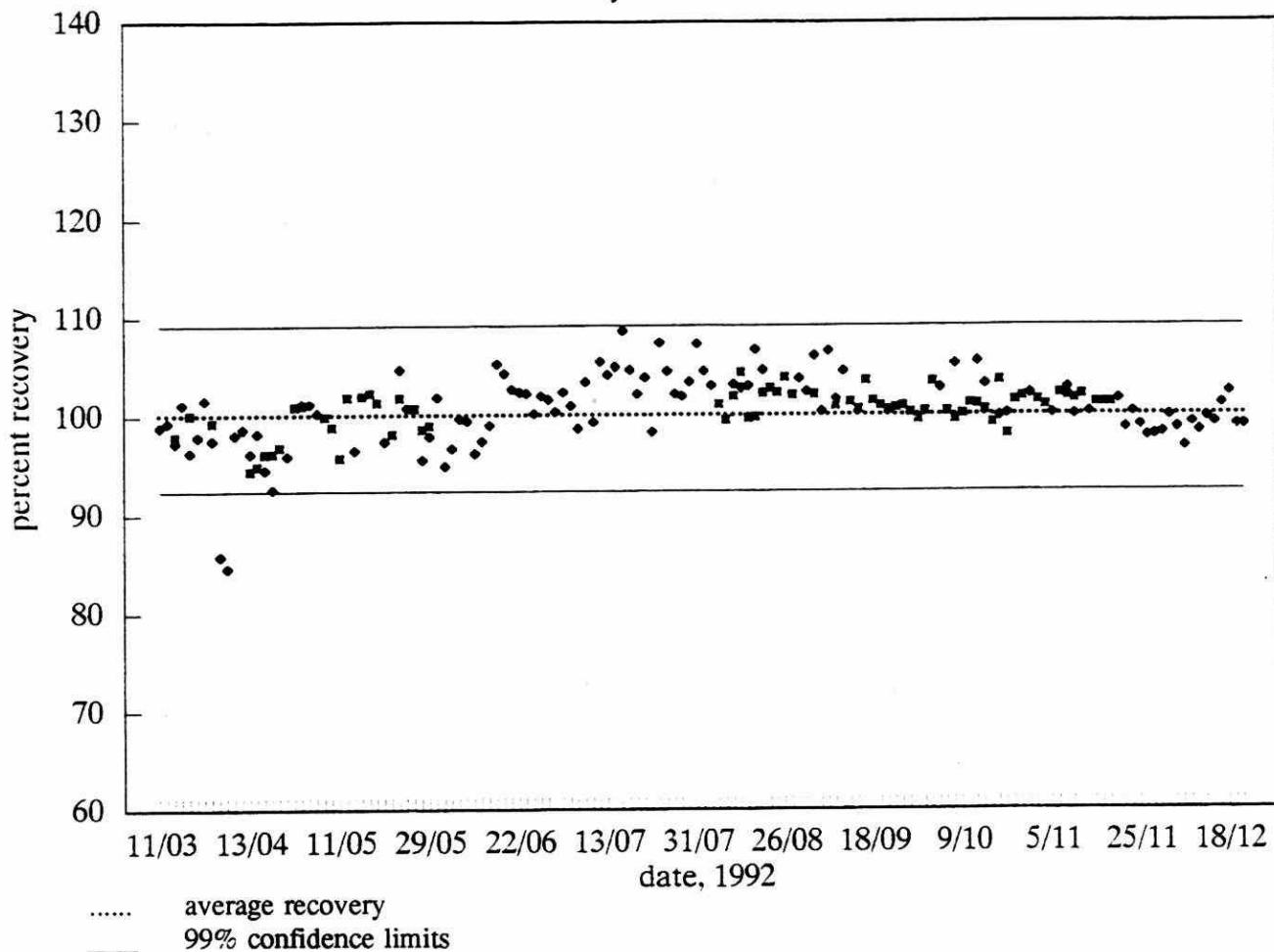
Performance Summary Table

January - December 1992

|                                    |            |
|------------------------------------|------------|
| Analyte                            | benzene    |
| True Concentration                 | 3.68 µg/L  |
| Number of Observations             | 172        |
| Within-run Rel. Standard Deviation | 1.1% (n=7) |
| Between-run Standard Deviation     | 1.7%       |
| Accuracy (% of expected)           | 100.2%     |

## 1,2-dichloroethane

recovery from fortified blank



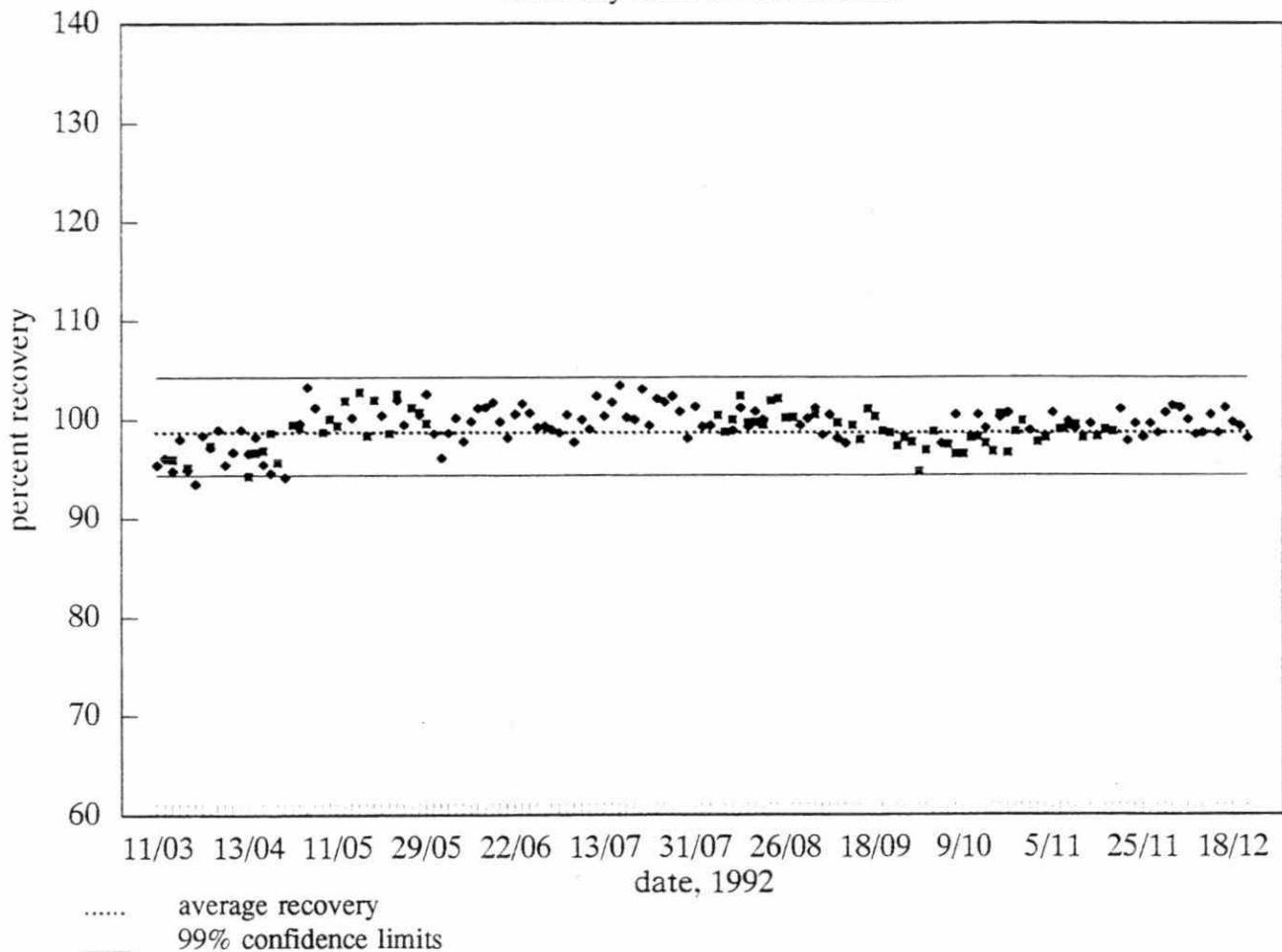
Performance Summary Table

January - December 1992

|                                    |                    |
|------------------------------------|--------------------|
| Analyte                            | 1,2-dichloroethane |
| True Concentration                 | 3.68 µg/L          |
| Number of Observations             | 172                |
| Within-run Rel. Standard Deviation | 2.4% (n=7)         |
| Between-run Standard Deviation     | 3.3%               |
| Accuracy (% of expected)           | 100.8%             |

## trichloroethene

recovery from fortified blank



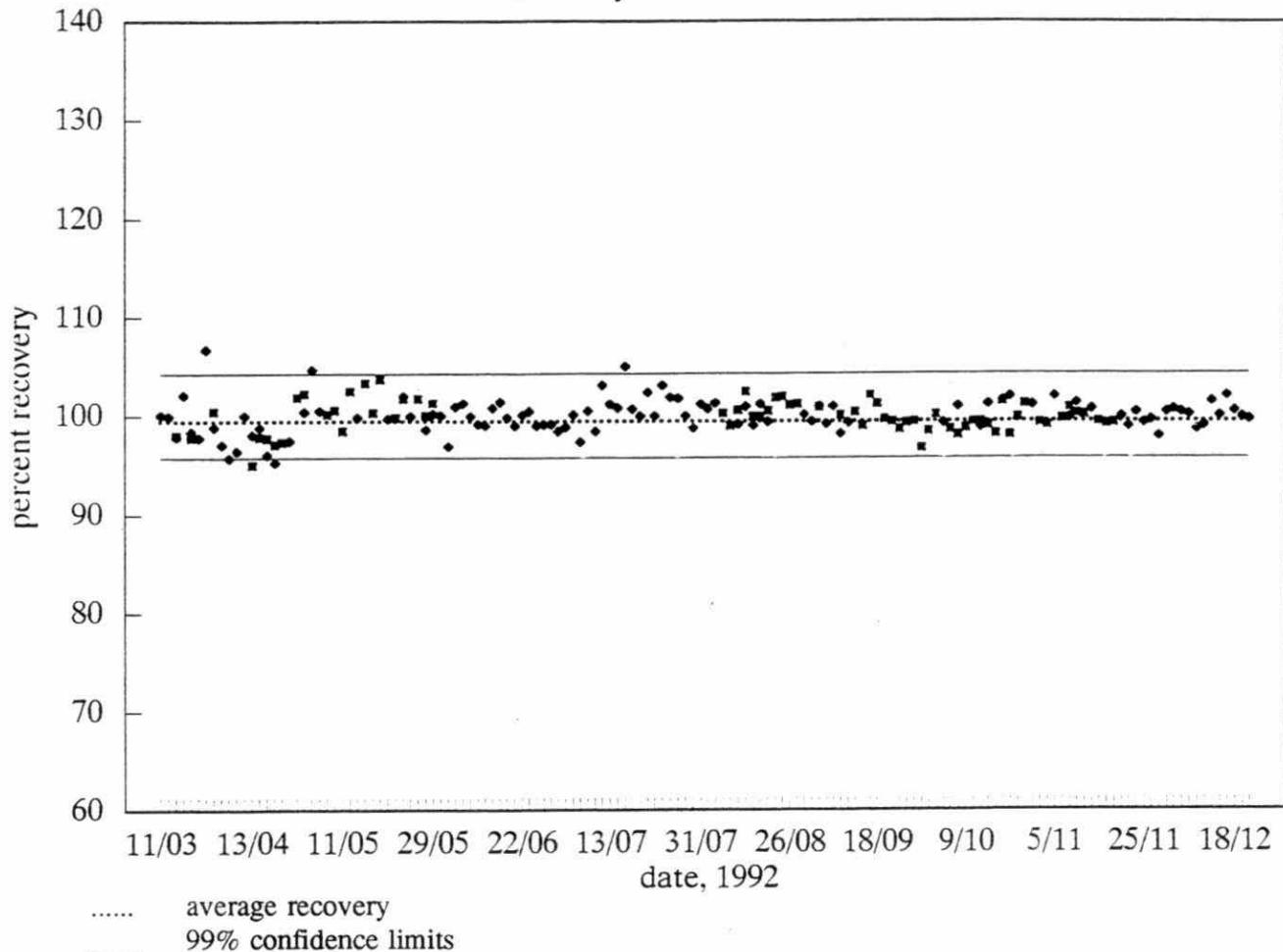
Performance Summary Table

January - December 1992

| Analyte                            | trichloroethene |
|------------------------------------|-----------------|
| True Concentration                 | 3.68 µg/L       |
| Number of Observations             | 172             |
| Within-run Rel. Standard Deviation | 1.3% (n=7)      |
| Between-run Standard Deviation     | 1.9%            |
| Accuracy (% of expected)           | 99.3%           |

## 1,2-dichloropropane

recovery from fortified blank



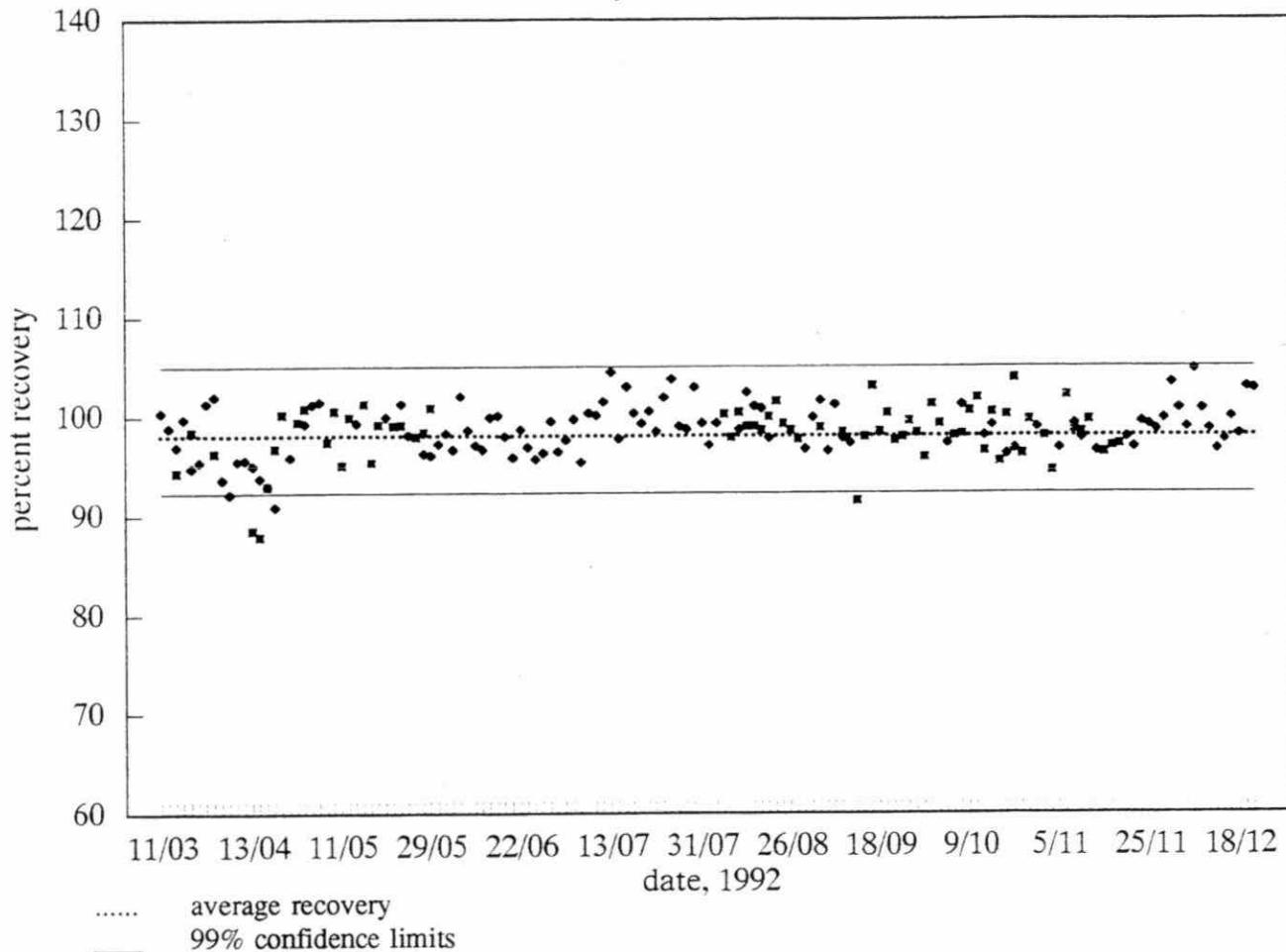
Performance Summary Table

January - December 1992

|                                    |                     |
|------------------------------------|---------------------|
| Analyte                            | 1,2-dichloropropane |
| True Concentration                 | 3.68 µg/L           |
| Number of Observations             | 172                 |
| Within-run Rel. Standard Deviation | 1.3% (n=7)          |
| Between-run Standard Deviation     | 1.7%                |
| Accuracy (% of expected)           | 100.0%              |

## bromodichloromethane

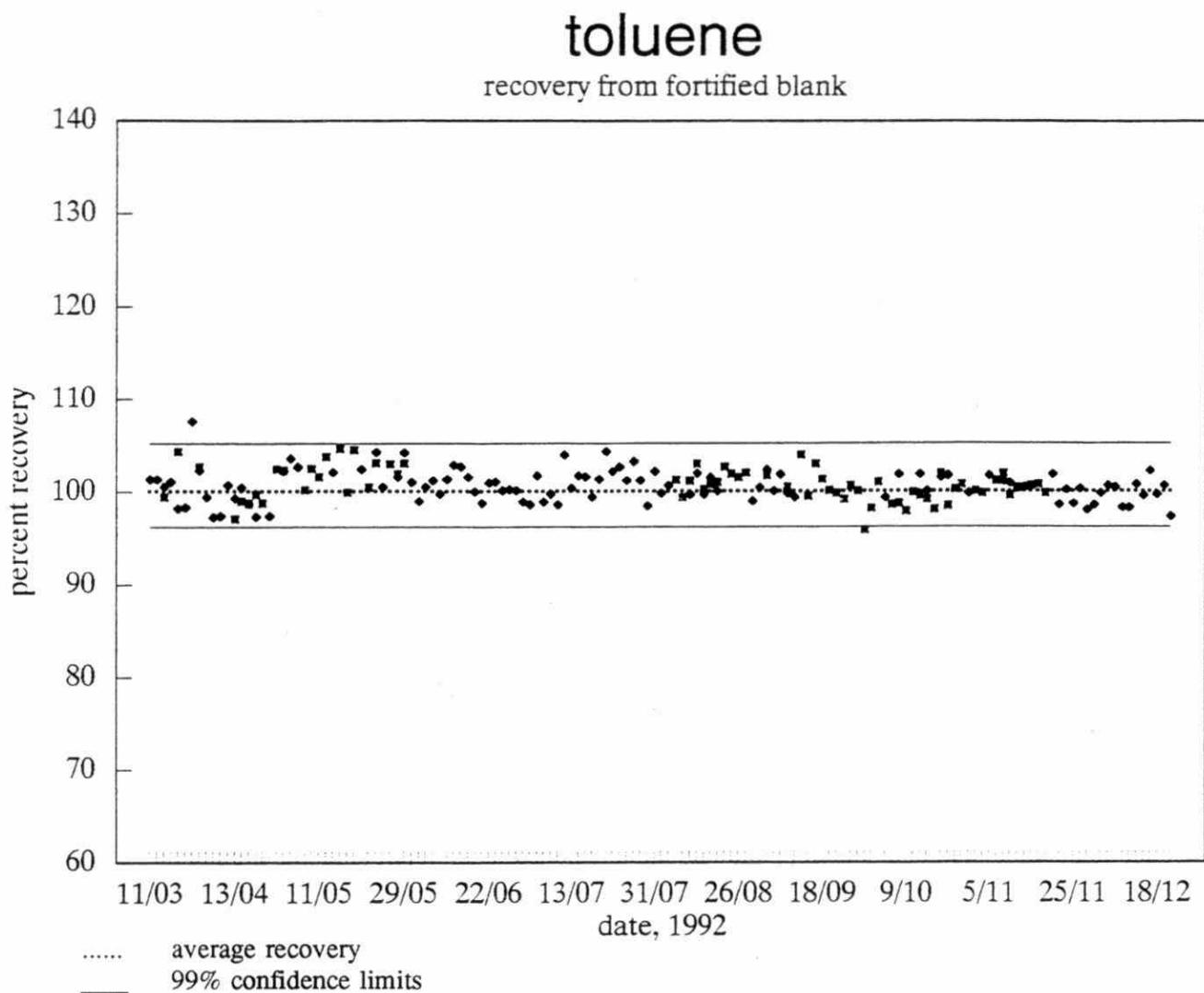
recovery from fortified blank



Performance Summary Table

January - December 1992

| Analyte                            | bromodichloromethane |
|------------------------------------|----------------------|
| True Concentration                 | 5.52 µg/L            |
| Number of Observations             | 172                  |
| Within-run Rel. Standard Deviation | 1.3% (n=7)           |
| Between-run Standard Deviation     | 2.5%                 |
| Accuracy (% of expected)           | 98.7%                |



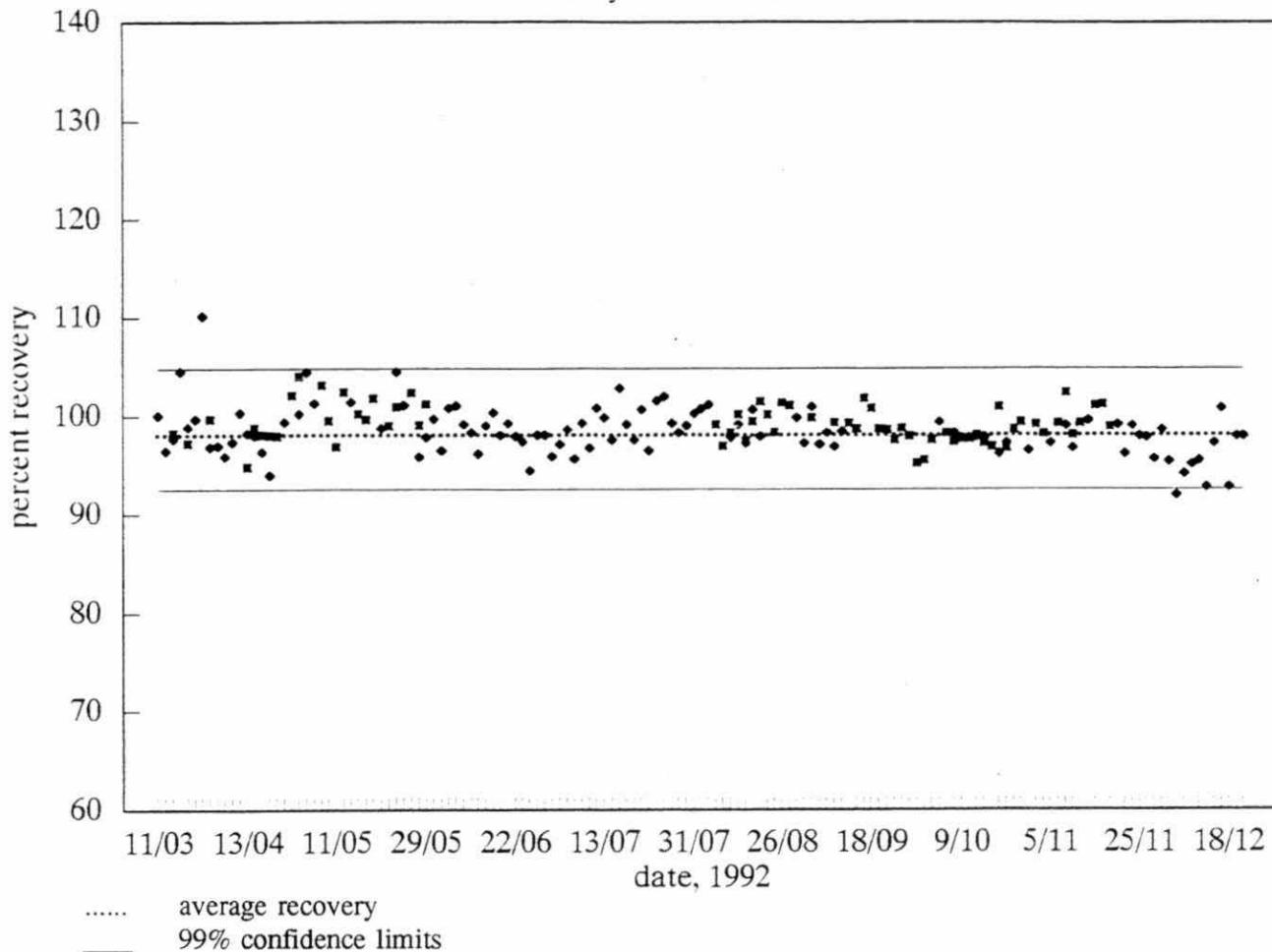
Performance Summary Table

January - December 1992

|                                    |            |
|------------------------------------|------------|
| Analyte                            | toluene    |
| True Concentration                 | 3.68 µg/L  |
| Number of Observations             | 172        |
| Within-run Rel. Standard Deviation | 1.0% (n=7) |
| Between-run Standard Deviation     | 1.8%       |
| Accuracy (% of expected)           | 100.8%     |

## 1,1,2-trichloroethane

recovery from fortified blank



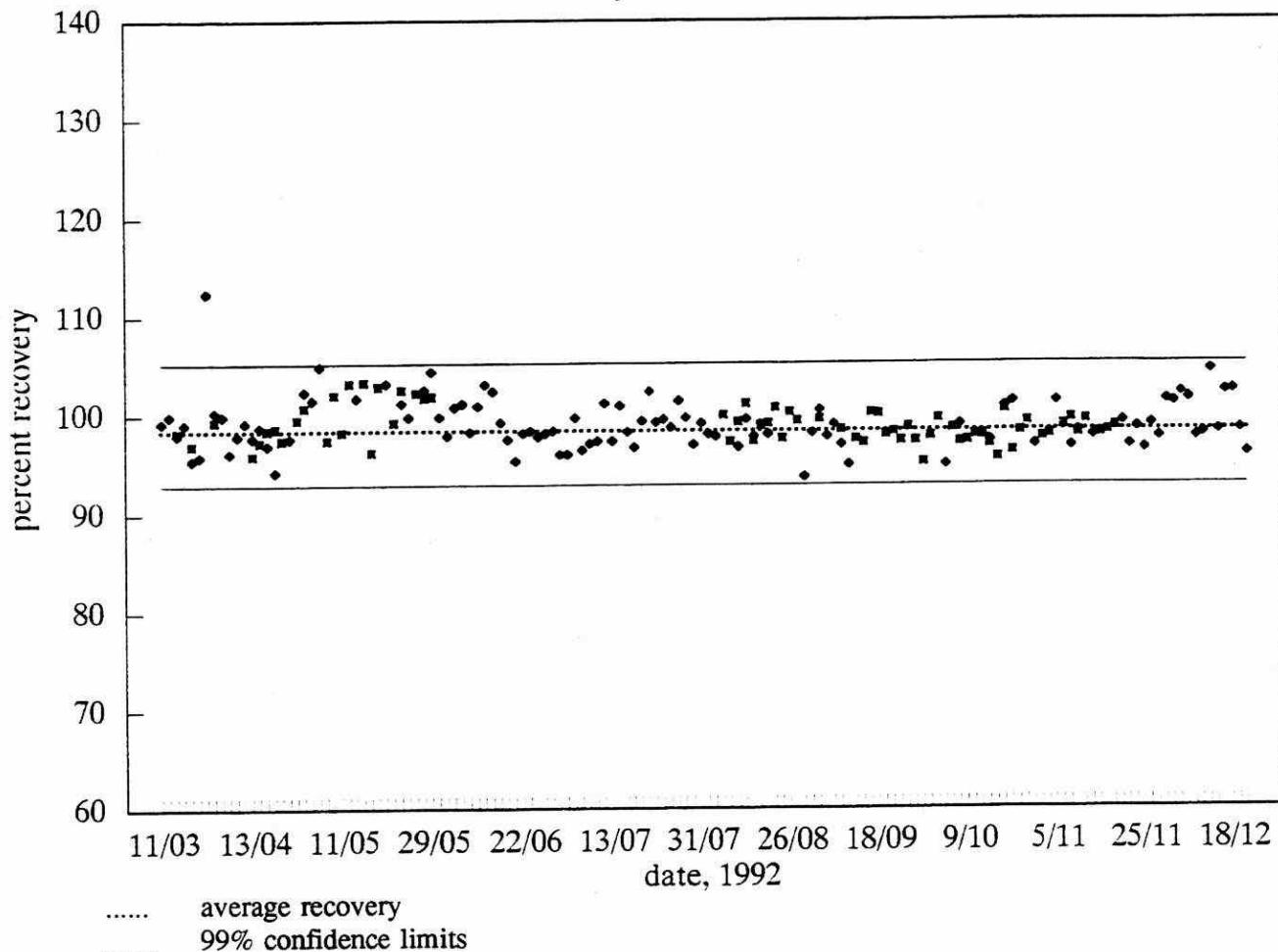
Performance Summary Table

January - December 1992

|                                    |                       |
|------------------------------------|-----------------------|
| Analyte                            | 1,1,2-trichloroethane |
| True Concentration                 | 2.01 µg/L, 3.68 µg/L  |
| Number of Observations             | 172                   |
| Within-run Rel. Standard Deviation | 1.2% (n=7)            |
| Between-run Standard Deviation     | 2.4%                  |
| Accuracy (% of expected)           | 99.1%                 |

## tetrachloroethene

recovery from fortified blank



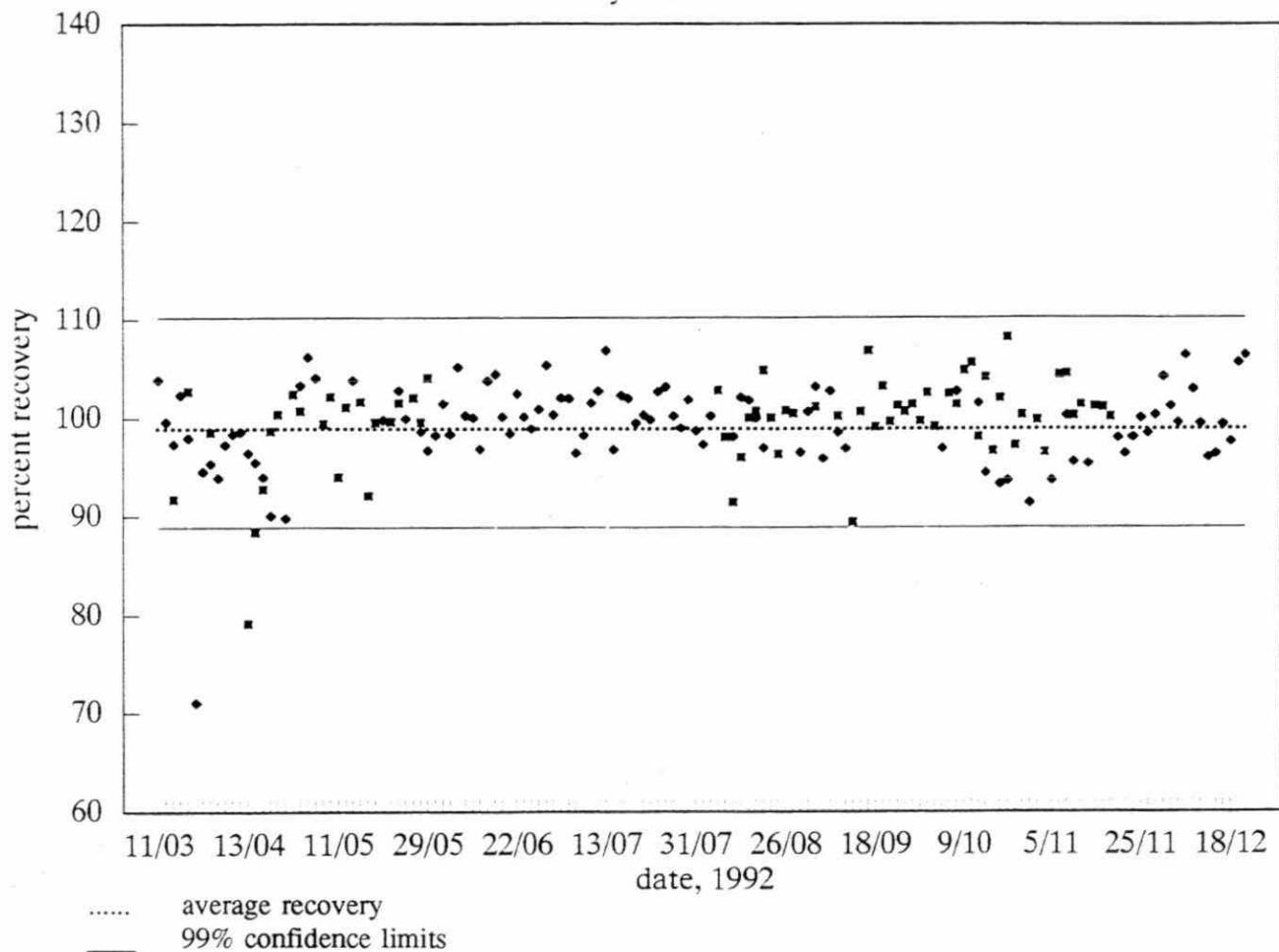
Performance Summary Table

January - December 1992

| Analyte                            | tetrachloroethene |
|------------------------------------|-------------------|
| True Concentration                 | 3.68 µg/L         |
| Number of Observations             | 172               |
| Within-run Rel. Standard Deviation | 1.1% (n=7)        |
| Between-run Standard Deviation     | 2.4%              |
| Accuracy (% of expected)           | 99.1%             |

## dibromochloromethane

recovery from fortified blank



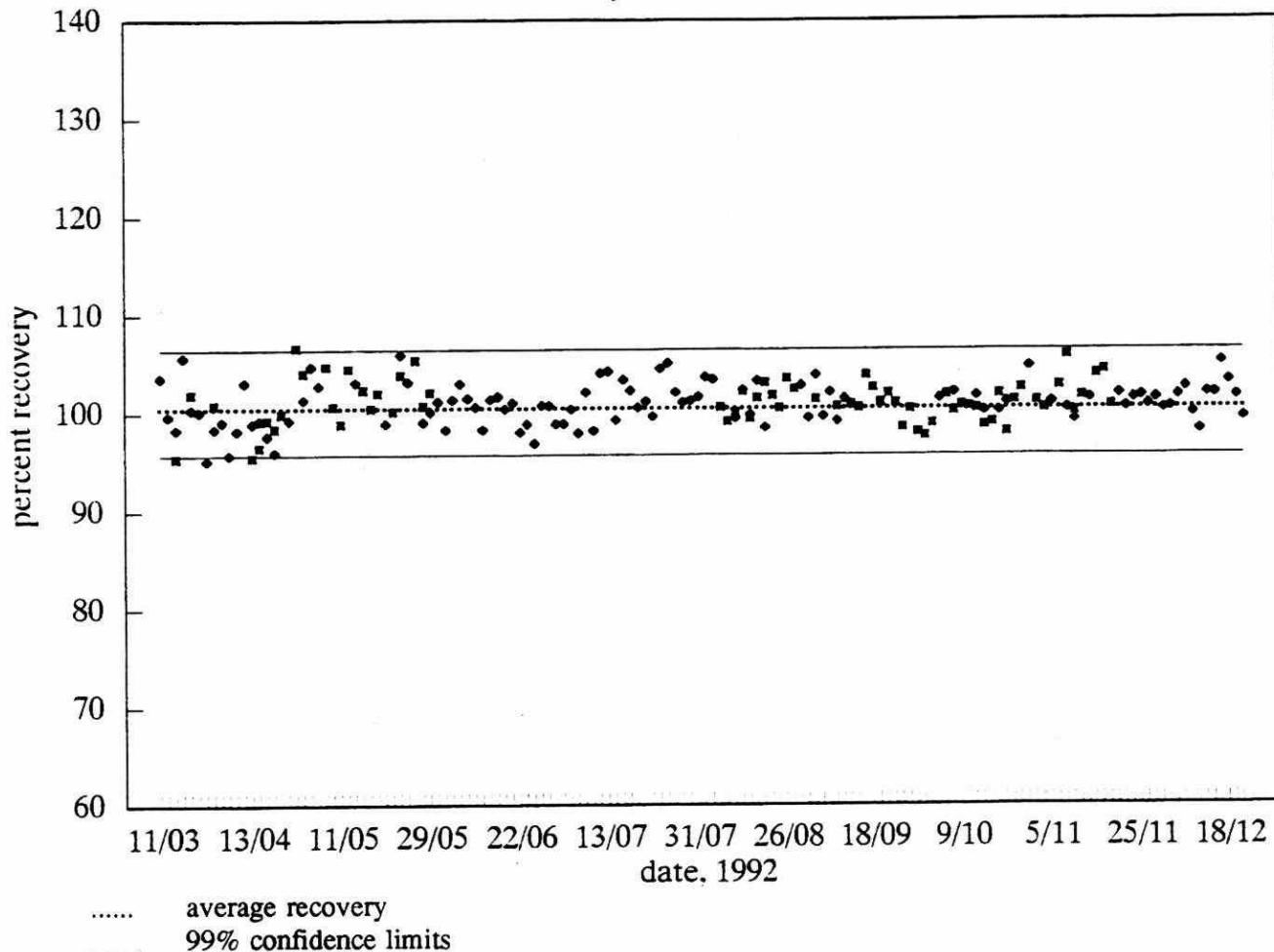
Performance Summary Table

January - December 1992

| Analyte                            | dibromochloromethane |
|------------------------------------|----------------------|
| True Concentration                 | 6.44 µg/L            |
| Number of Observations             | 172                  |
| Within-run Rel. Standard Deviation | 2.5% (n=7)           |
| Between-run Standard Deviation     | 4.2%                 |
| Accuracy (% of expected)           | 99.6%                |

## 1,2-dibromoethane

recovery from fortified blank



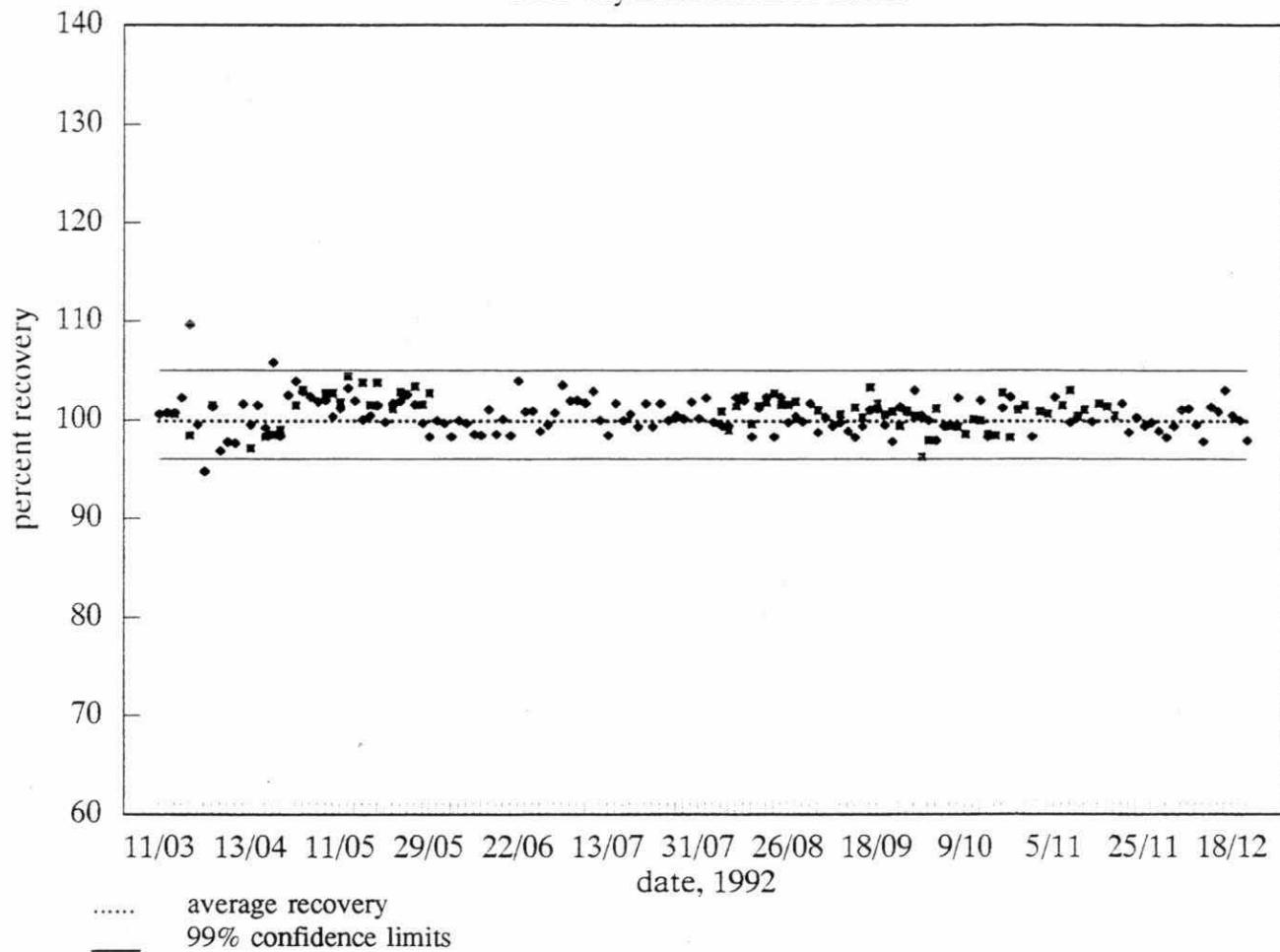
Performance Summary Table

January - December 1992

|                                    |                   |
|------------------------------------|-------------------|
| Analyte                            | 1,2-dibromoethane |
| True Concentration                 | 5.52 µg/L         |
| Number of Observations             | 172               |
| Within-run Rel. Standard Deviation | 2.0% (n=7)        |
| Between-run Standard Deviation     | 2.1%              |
| Accuracy (% of expected)           | 101.1%            |

## chlorobenzene

recovery from fortified blank

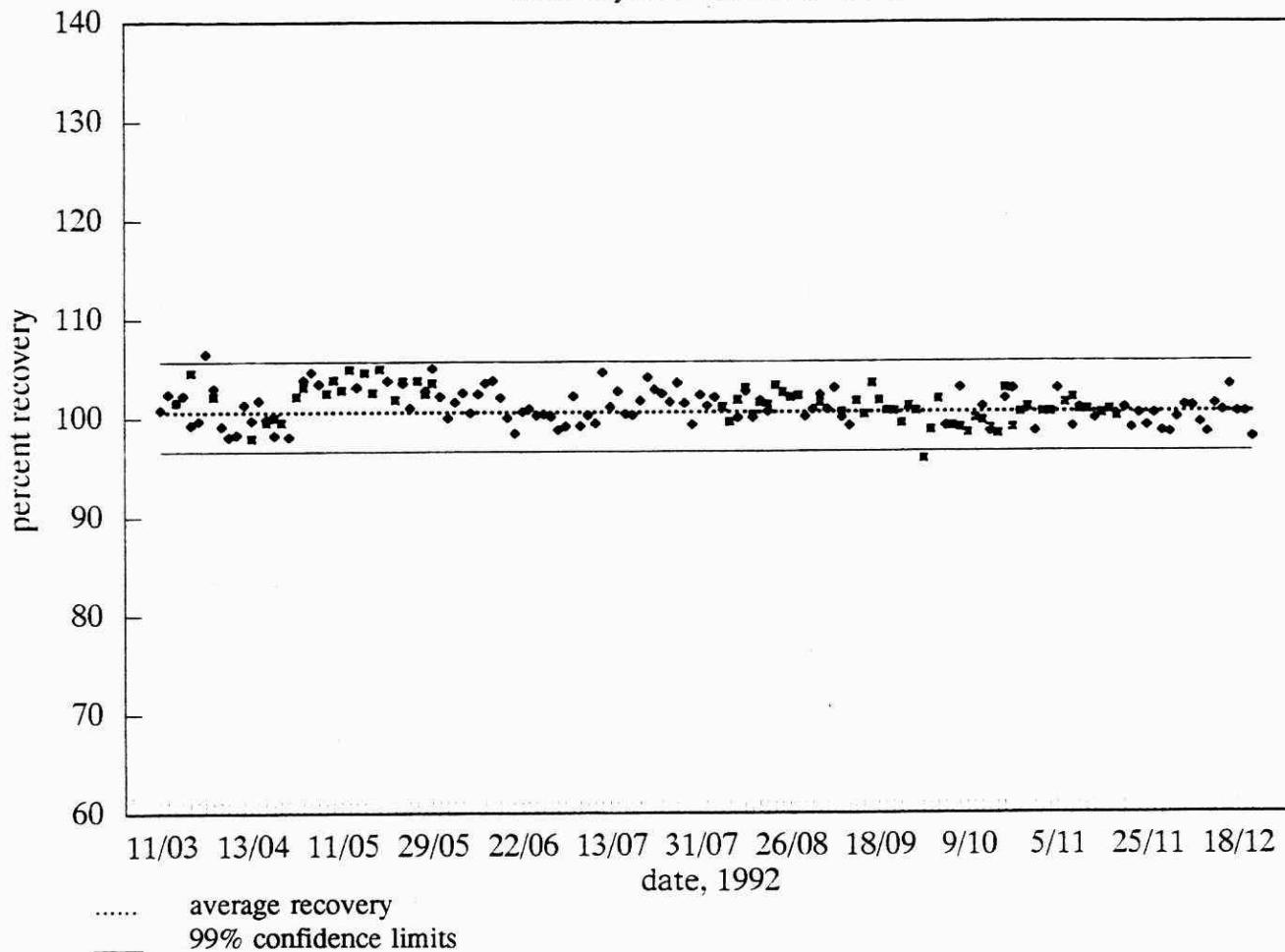


Performance Summary Table

January - December 1992

| Analyte                            | chlorobenzene |
|------------------------------------|---------------|
| True Concentration                 | 4.6 µg/L      |
| Number of Observations             | 172           |
| Within-run Rel. Standard Deviation | 0.9% (n=7)    |
| Between-run Standard Deviation     | 1.8%          |
| Accuracy (% of expected)           | 100.5%        |

**ethylbenzene**  
recovery from fortified blank

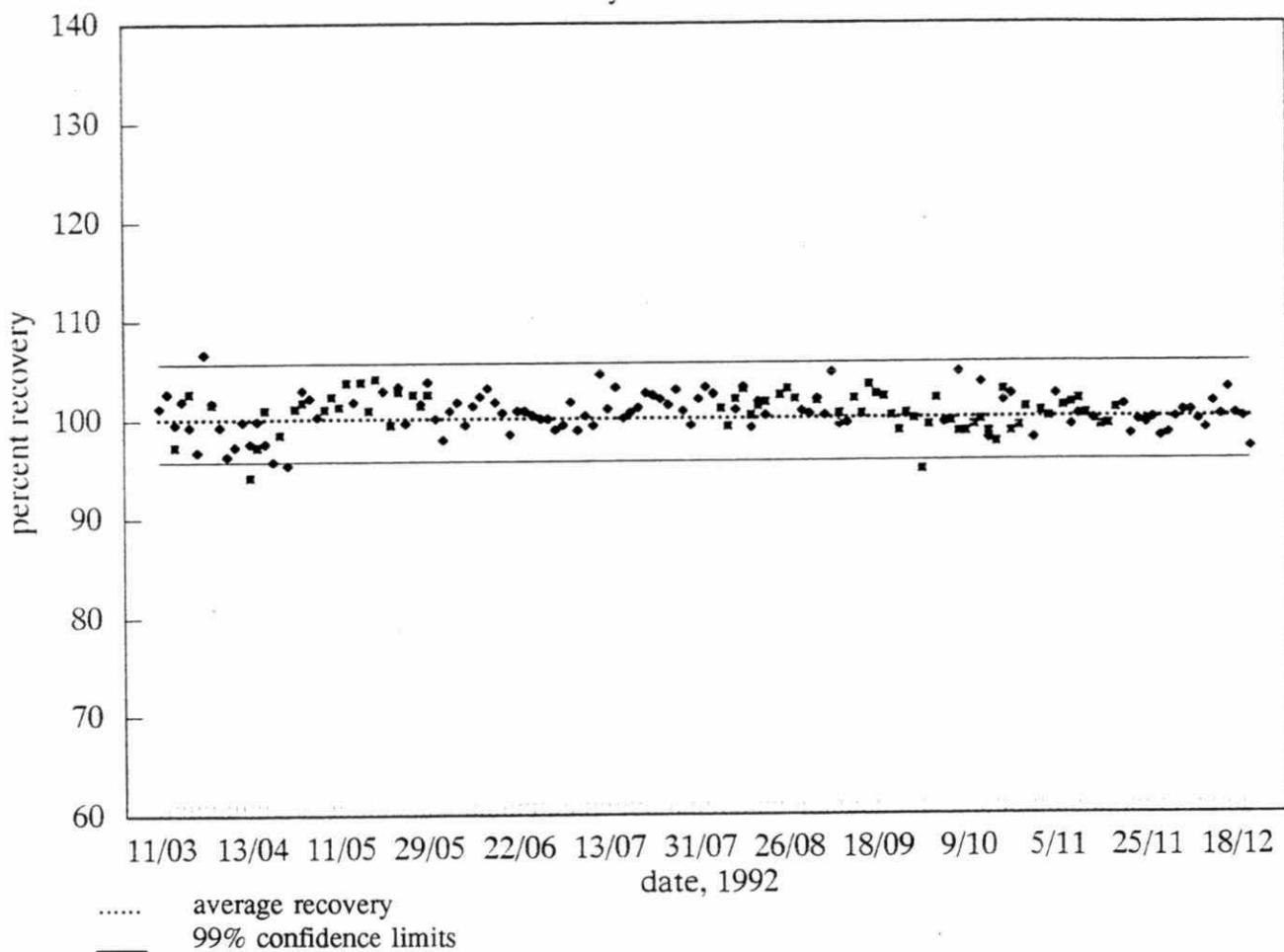


**Performance Summary Table**

January - December 1992

|                                    |              |
|------------------------------------|--------------|
| Analyte                            | ethylbenzene |
| True Concentration                 | 3.68 µg/L    |
| Number of Observations             | 172          |
| Within-run Rel. Standard Deviation | 0.9% (n=7)   |
| Between-run Standard Deviation     | 1.8%         |
| Accuracy (% of expected)           | 101.2%       |

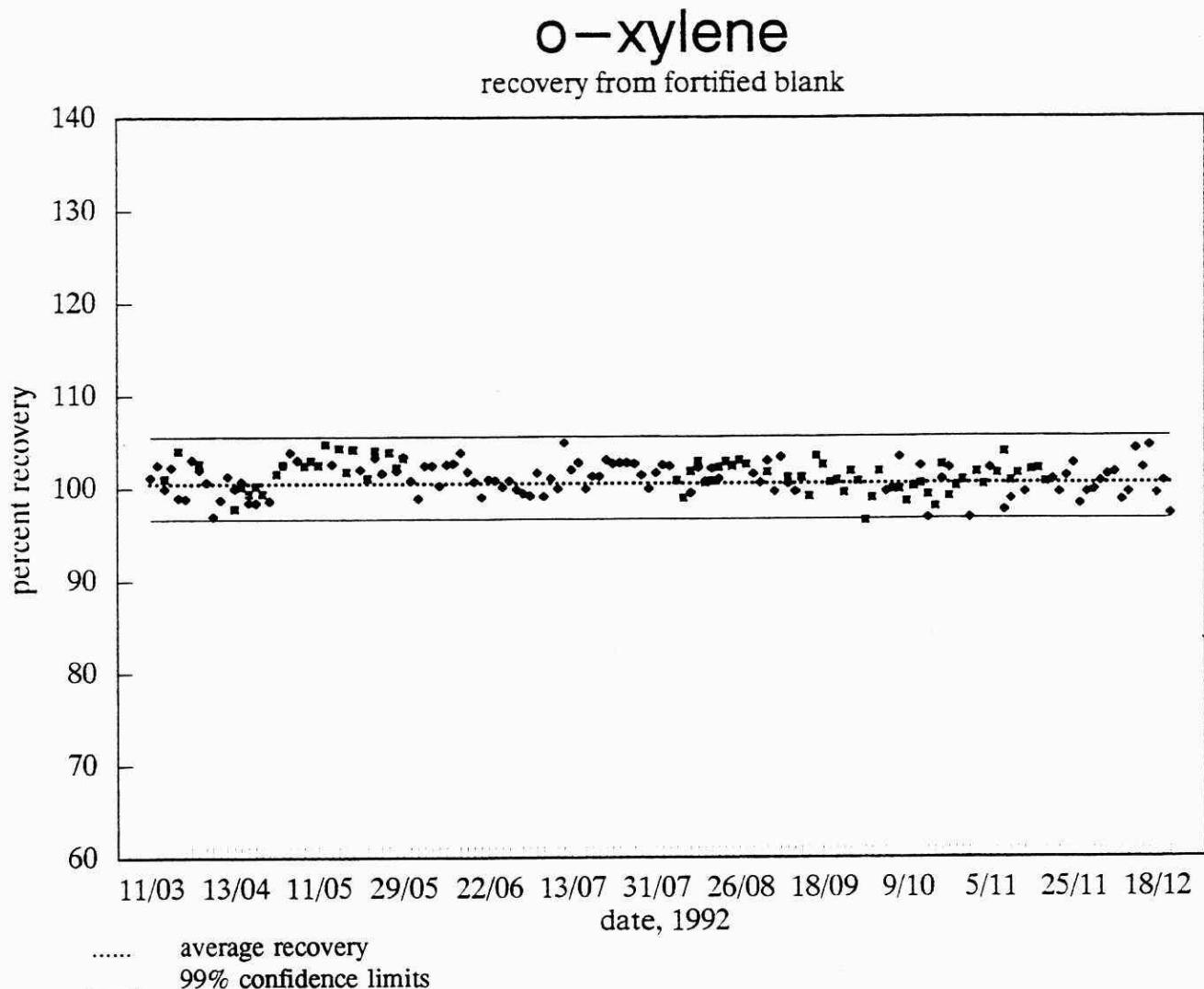
**m/p-xylene**  
recovery from fortified blank



**Performance Summary Table**

January - December 1992

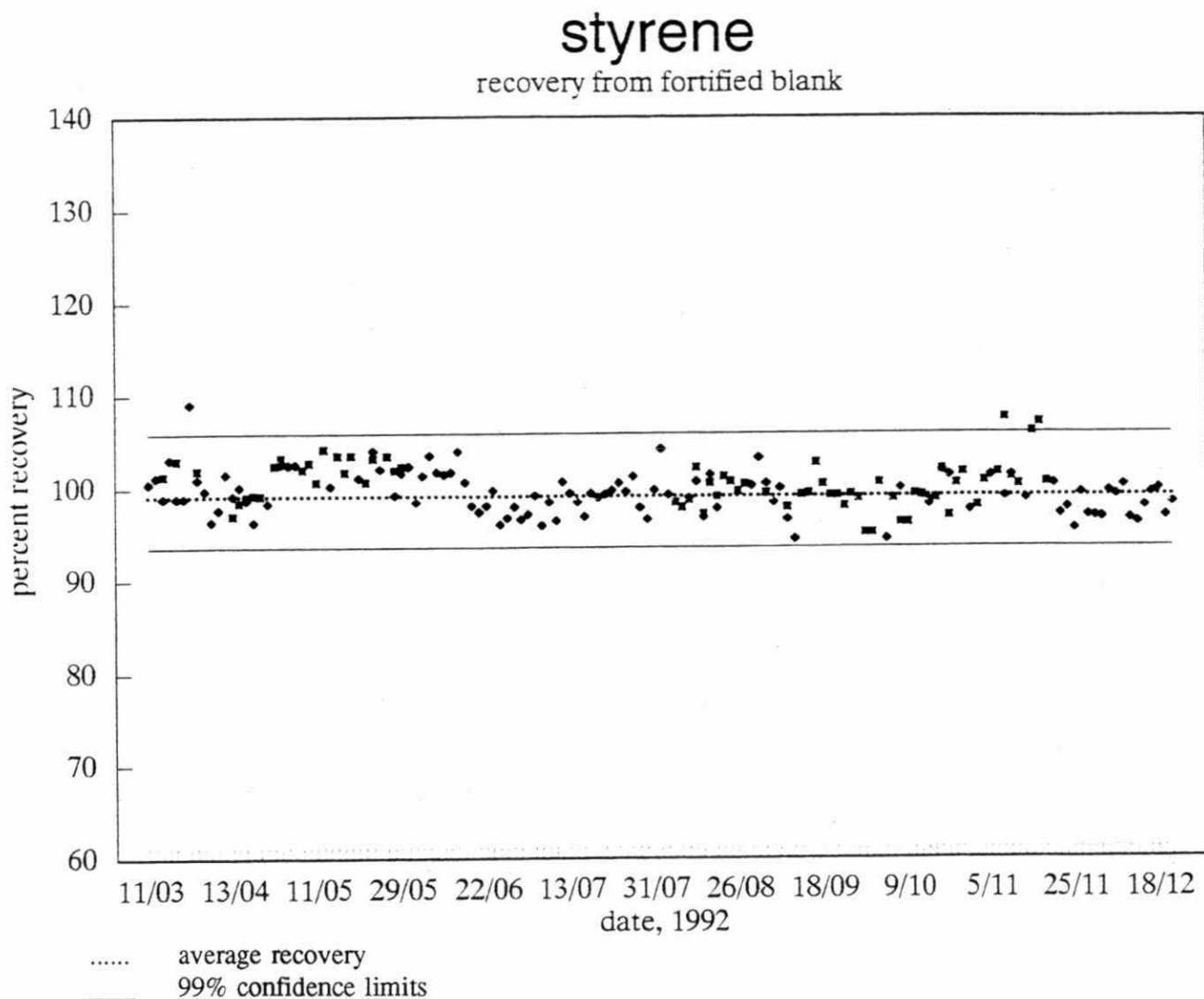
| Analyte                            | m/p-xylene |
|------------------------------------|------------|
| True Concentration                 | 3.68 µg/L  |
| Number of Observations             | 171        |
| Within-run Rel. Standard Deviation | 0.8% (n=7) |
| Between-run Standard Deviation     | 1.9%       |
| Accuracy (% of expected)           | 100.7%     |



Performance Summary Table

January - December 1992

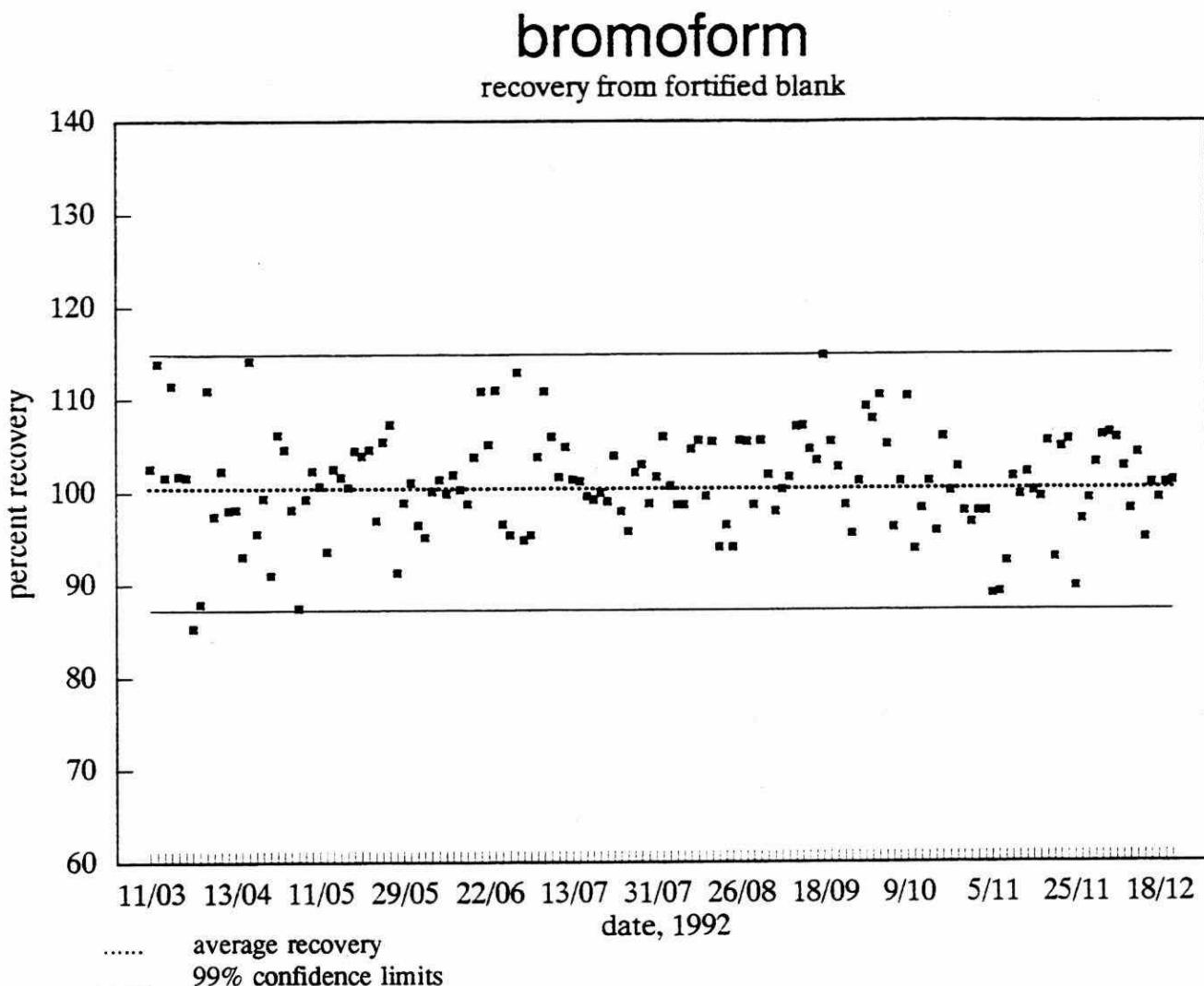
|                                    |            |
|------------------------------------|------------|
| Analyte                            | o-xylene   |
| True Concentration                 | 3.68 µg/L  |
| Number of Observations             | 172        |
| Within-run Rel. Standard Deviation | 1.3% (n=7) |
| Between-run Standard Deviation     | 1.8%       |
| Accuracy (% of expected)           | 101.1%     |



Performance Summary Table

January - December 1992

|                                    |            |
|------------------------------------|------------|
| Analyte                            | styrene    |
| True Concentration                 | 3.68 µg/L  |
| Number of Observations             | 172        |
| Within-run Rel. Standard Deviation | 1.2% (n=7) |
| Between-run Standard Deviation     | 2.4%       |
| Accuracy (% of expected)           | 99.7%      |



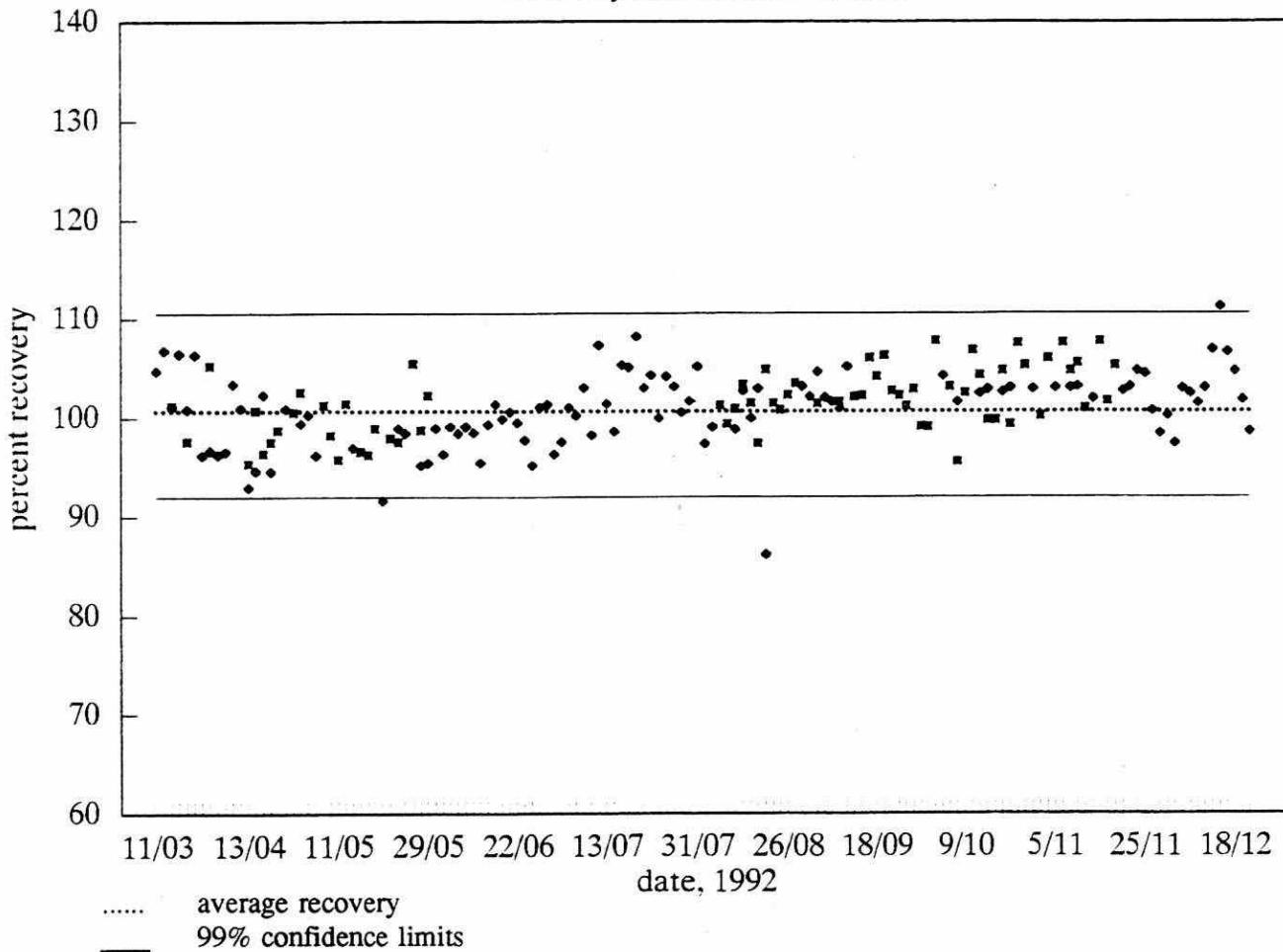
Performance Summary Table

January - December 1992

|                                    |            |
|------------------------------------|------------|
| Analyte                            | bromoform  |
| True Concentration                 | 7.36 µg/L  |
| Number of Observations             | 172        |
| Within-run Rel. Standard Deviation | 4.4% (n=7) |
| Between-run Standard Deviation     | 6%         |
| Accuracy (% of expected)           | 100%       |

## 1,1,2,2-tetrachloroethane

recovery from fortified blank



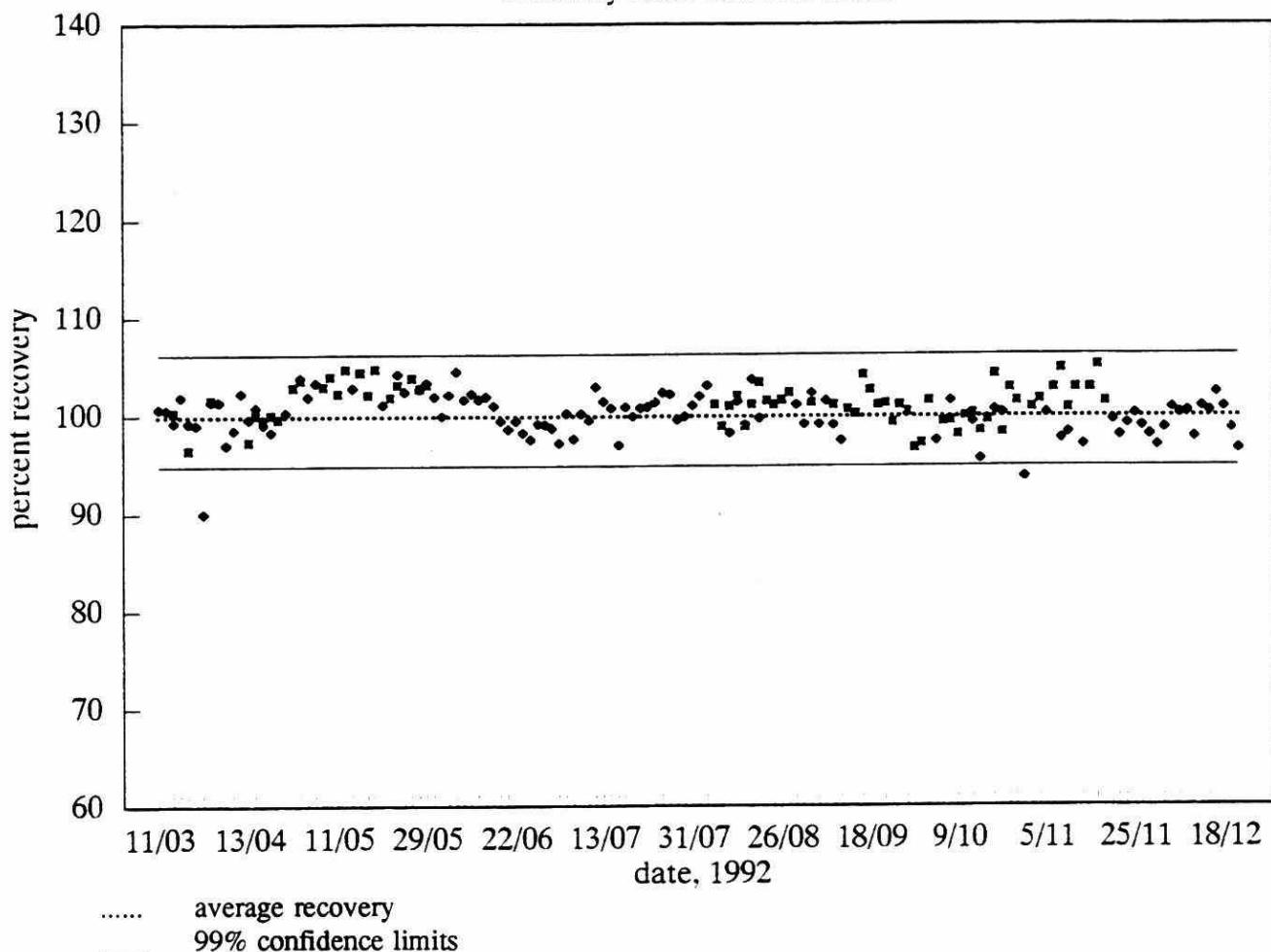
Performance Summary Table

January - December 1992

| Analyte                            | 1,1,2,2-tetrachloroethane |
|------------------------------------|---------------------------|
| True Concentration                 | 5.52 µg/L                 |
| Number of Observations             | 172                       |
| Within-run Rel. Standard Deviation | 1.7% (n=7)                |
| Between-run Standard Deviation     | 3.7%                      |
| Accuracy (% of expected)           | 101.3%                    |

## 1,3-dichlorobenzene

recovery from fortified blank



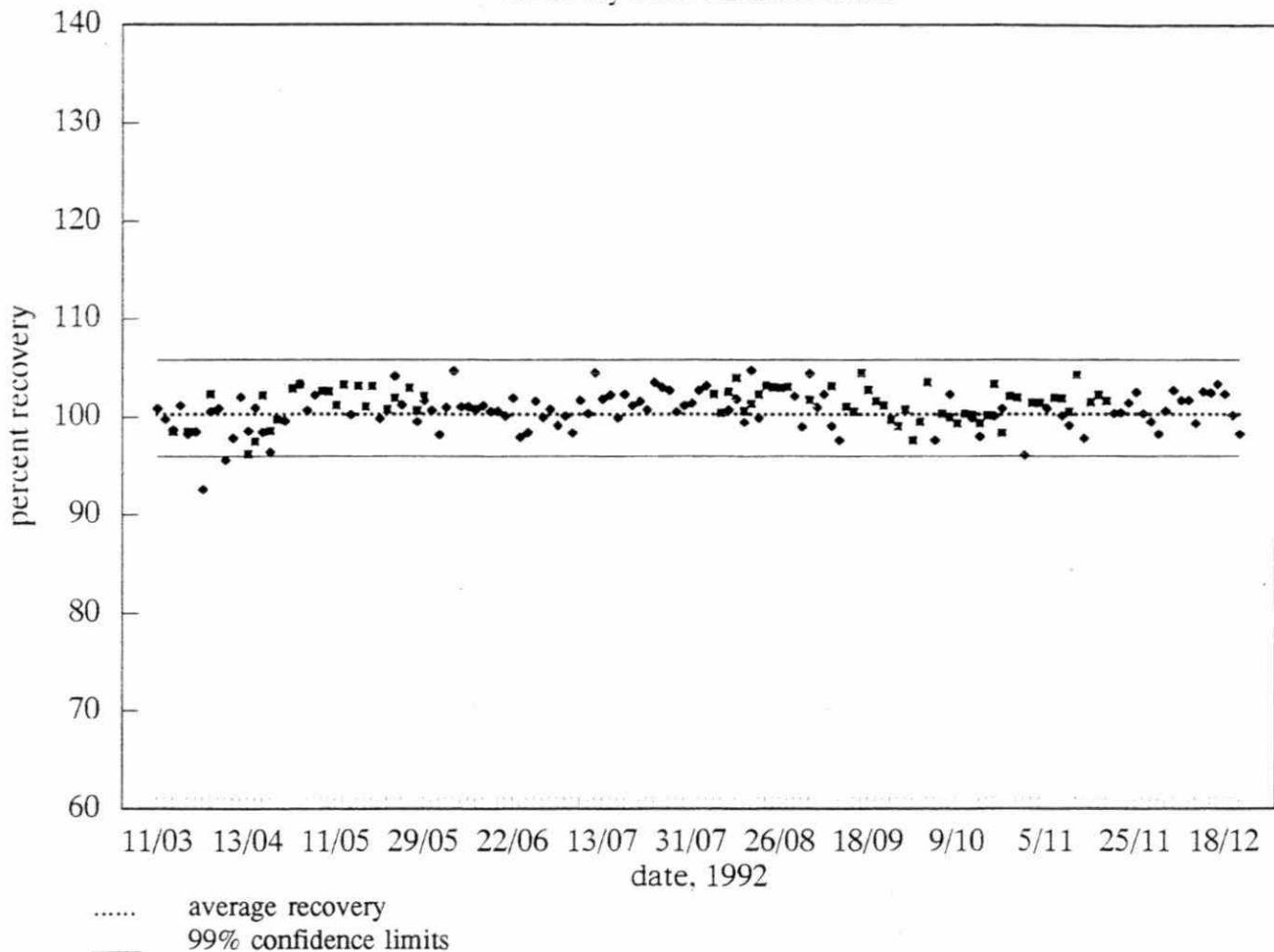
Performance Summary Table

January - December 1992

|                                    |                     |
|------------------------------------|---------------------|
| Analyte                            | 1,3-dichlorobenzene |
| True Concentration                 | 5.52 µg/L           |
| Number of Observations             | 172                 |
| Within-run Rel. Standard Deviation | 0.8% (n=7)          |
| Between-run Standard Deviation     | 2.2%                |
| Accuracy (% of expected)           | 100.5%              |

## 1,4-dichlorobenzene

recovery from fortified blank



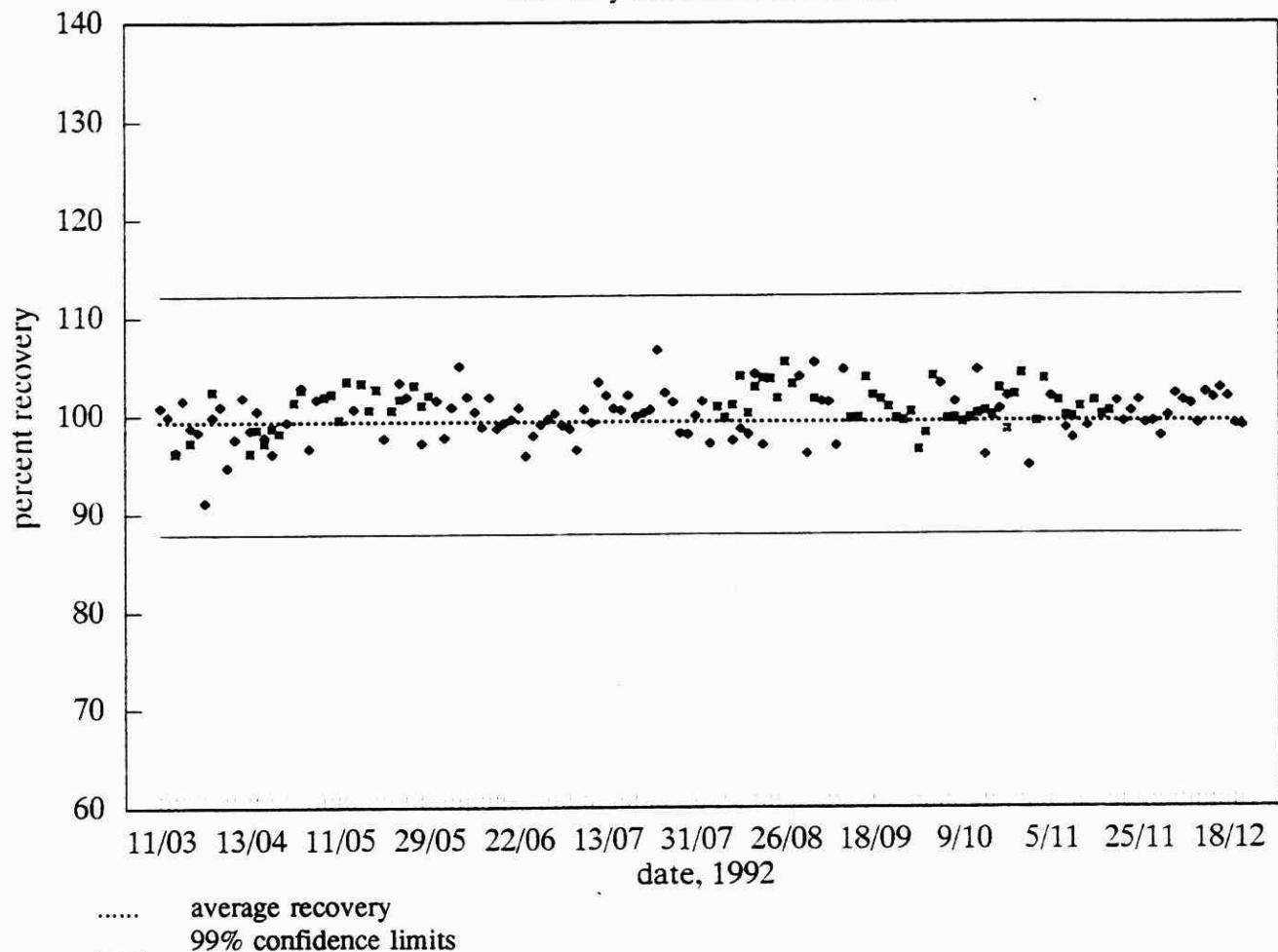
Performance Summary Table

January - December 1992

|                                    |                     |
|------------------------------------|---------------------|
| Analyte                            | 1,4-dichlorobenzene |
| True Concentration                 | 5.52 µg/L           |
| Number of Observations             | 172                 |
| Within-run Rel. Standard Deviation | 0.5% (n=7)          |
| Between-run Standard Deviation     | 1.9%                |
| Accuracy (% of expected)           | 100.9%              |

## 1,2-dichlorobenzene

recovery from fortified blank



Performance Summary Table

January - December 1992

|                                    |                     |
|------------------------------------|---------------------|
| Analyte                            | 1,2-dichlorobenzene |
| True Concentration                 | 5.52 µg/L           |
| Number of Observations             | 172                 |
| Within-run Rel. Standard Deviation | 0.8% (n=7)          |
| Between-run Standard Deviation     | 4.8%                |
| Accuracy (% of expected)           | 100.0%              |

**METHOD CODE :** OPTM-E3237A

**METHOD TITLE:** The Determination of Trihalomethanes in Water by Purge-and-Trap/Gas Chromatography

**LABORATORY :** Priority Pollutants Unit

**SUPERVISOR :** Dr. W. Berg

**SAMPLE TYPE :** raw and treated drinking water

**PRINCIPLE OF THE METHOD :**

Trihalomethanes are purged from an aqueous sample onto an adsorption trap, and subsequently, thermally desorbed onto a gas chromatographic capillary column. After separation, the organics are identified and quantified by Hall electrolytic conductivity detector.

| <b>PARAMETERS MEASURED :</b> | <b>LIS TEST CODE</b> | <b>W ( µg/L )</b> | <b>T ( µg/L )</b> |
|------------------------------|----------------------|-------------------|-------------------|
| Chloroform                   | X1005J               | 0.5               | 5.0               |
| Bromodichloromethane         | X1010J               | 0.2               | 2.0               |
| Dibromochloromethane         | X1011J               | 0.2               | 2.0               |
| Bromoform                    | X1015J               | 0.2               | 2.0               |
| Total THM's                  | X2TTHM               | 0.5               | 5.0               |

**REPORTING FORMAT :**

Results are reported in parts per billion (µg/L) rounded off to the closest increment of W and up to maximum of three significant figures.

**QUALITY CONTROL :**

The routine quality control samples are designed to verify absence of potential contamination ( method blanks ) and to monitor validity of calibration ( calibration solutions ) and the agreement with the established method precision and accuracy ( laboratory replicate samples, blank fortified with a certified solution ).

The results for the analysis of calibration solution and the analysis of reference material have their control limits statistically derived.

**REMARKS :** In addition to the intra-laboratory method control, the performance of the method was examined through performance audit samples program organized by LSB Quality Management Office.

List of Performance Charts : Chloroform ( recovery from fortified blank )  
Bromodichloromethane ( recovery from fortified blank )  
Dibromochloromethane ( recovery from fortified blank )  
Bromoform ( recovery from fortified blank )

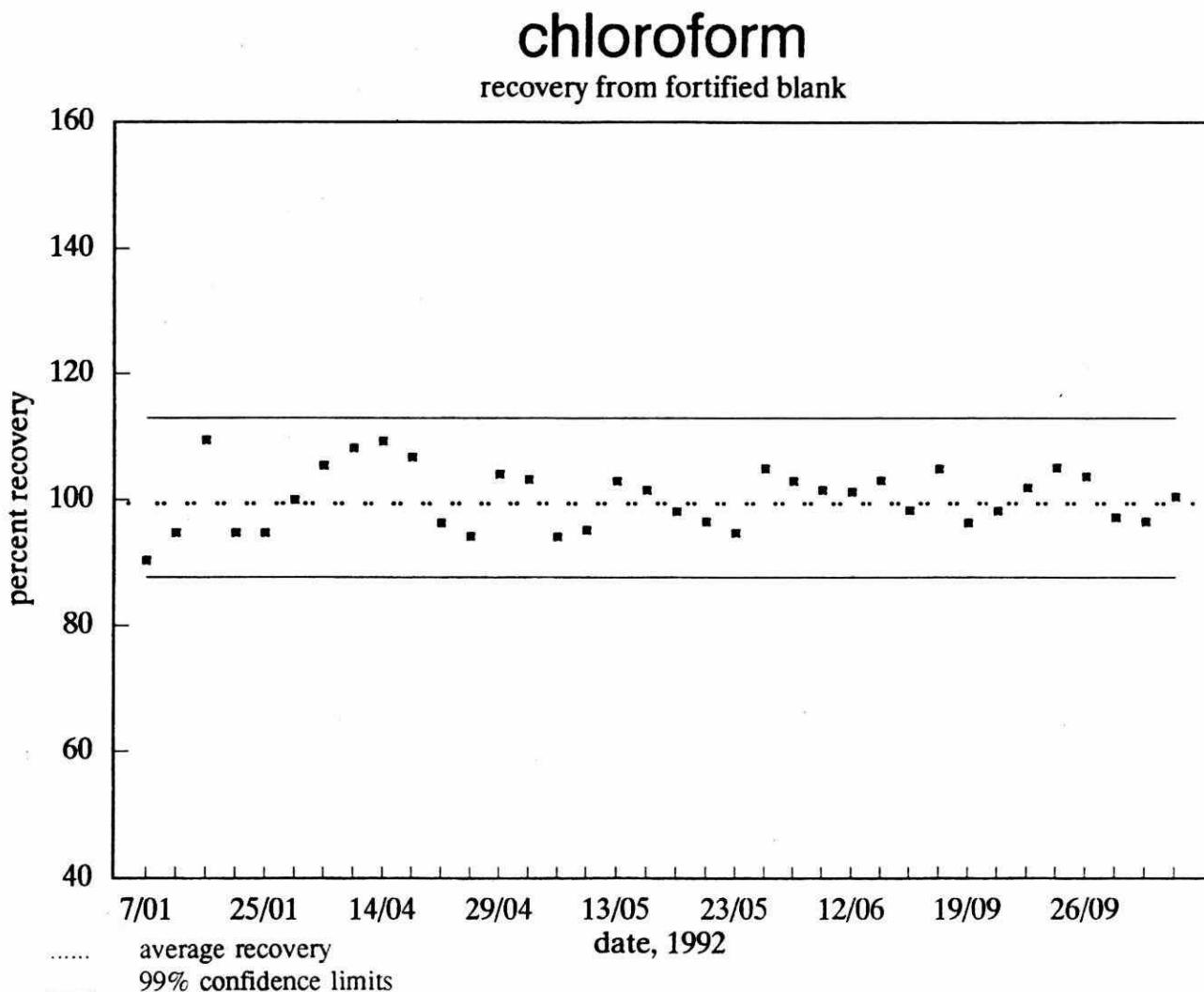
List of Performance Tables : Method Blanks Summary  
Chloroform  
Bromodichloromethane  
Dibromochloromethane  
Bromoform

Method Blanks Summary

January 1992 - December 1992

| Analyte              | Number of Observations | Average Concentration ( µg/L ) | Standard Deviation ( µg/L ) |
|----------------------|------------------------|--------------------------------|-----------------------------|
| chloroform           | 104                    | 0.09                           | 0.24                        |
| bromodichloromethane | 104                    | 0.02                           | 0.07                        |
| dibromochloromethane | 104                    | 0.004                          | 0.027                       |
| bromoform            | 104                    | ND ( 0.3 )                     |                             |
| THM's - total        | 104                    | 0.12                           | 0.30                        |

ND ... Not detected. Detection limit in µg/L given in brackets ( ).



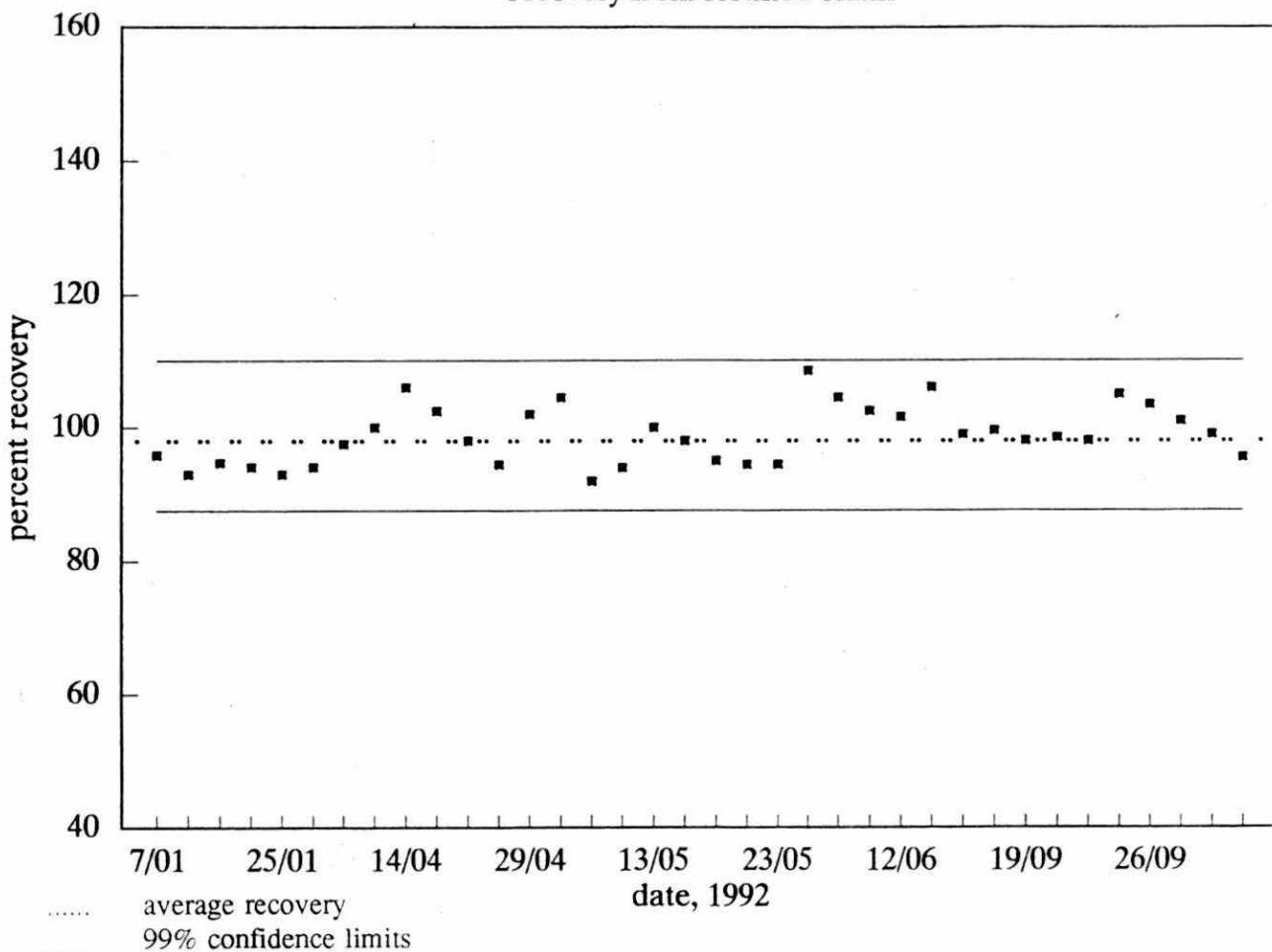
Performance Summary Table

January - December 1992

|                                    |             |
|------------------------------------|-------------|
| Analyte                            | chloroform  |
| True Concentration                 | 100 µg/L    |
| Number of Observations             | 36          |
| Within-run Rel. Standard Deviation | 2.4% (n=13) |
| Between-run Standard Deviation     | 4.9%        |
| Accuracy (% of expected)           | 100.3%      |

## bromodichloromethane

recovery from fortified blank



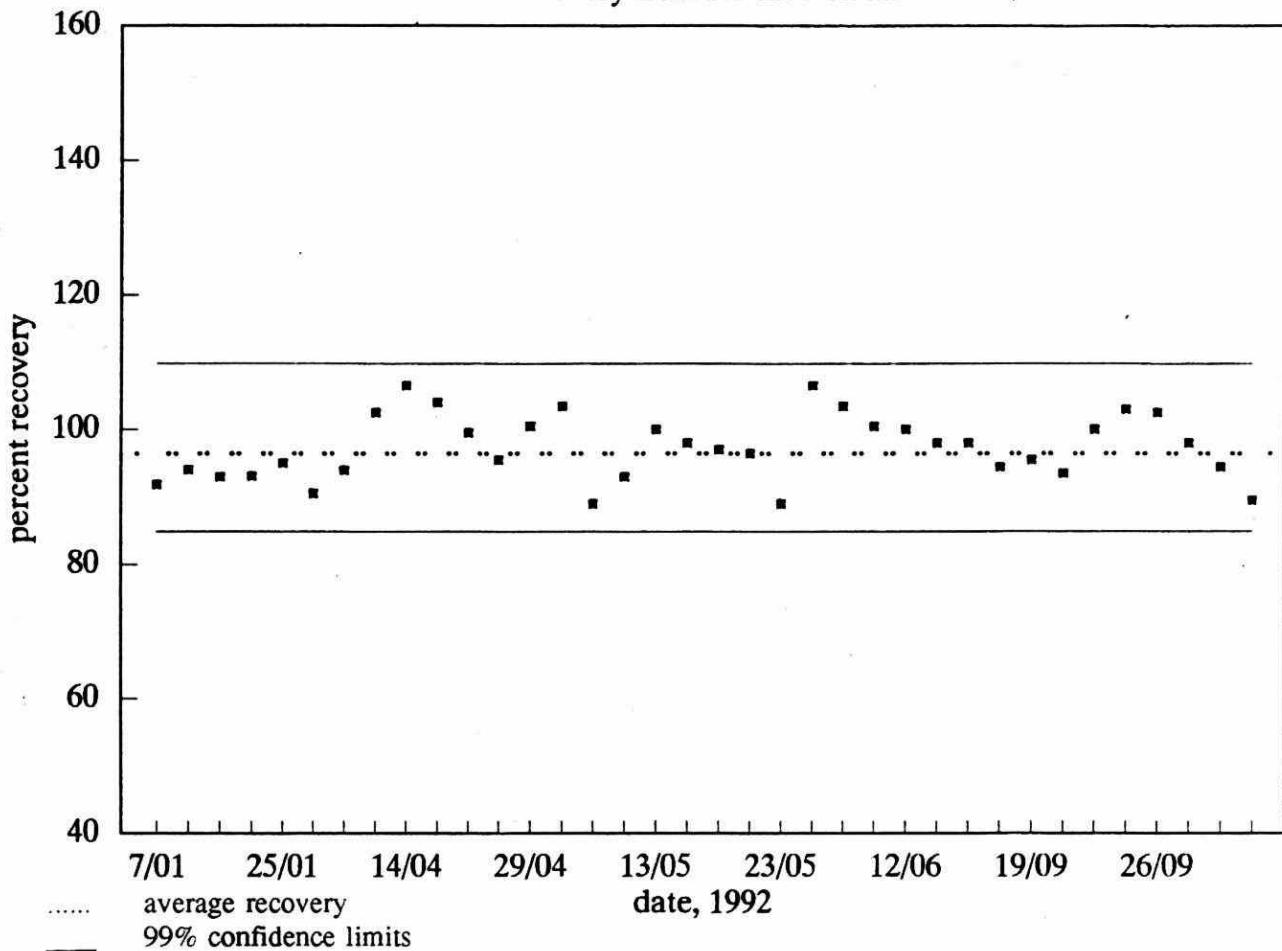
Performance Summary Table

January - December 1992

|                                    |                      |
|------------------------------------|----------------------|
| Analyte                            | bromodichloromethane |
| True Concentration                 | 20 µg/L              |
| Number of Observations             | 36                   |
| Within-run Rel. Standard Deviation | 1.9% (n=13)          |
| Between-run Standard Deviation     | 4.4%                 |
| Accuracy (% of expected)           | 98.8%                |

## dibromochloromethane

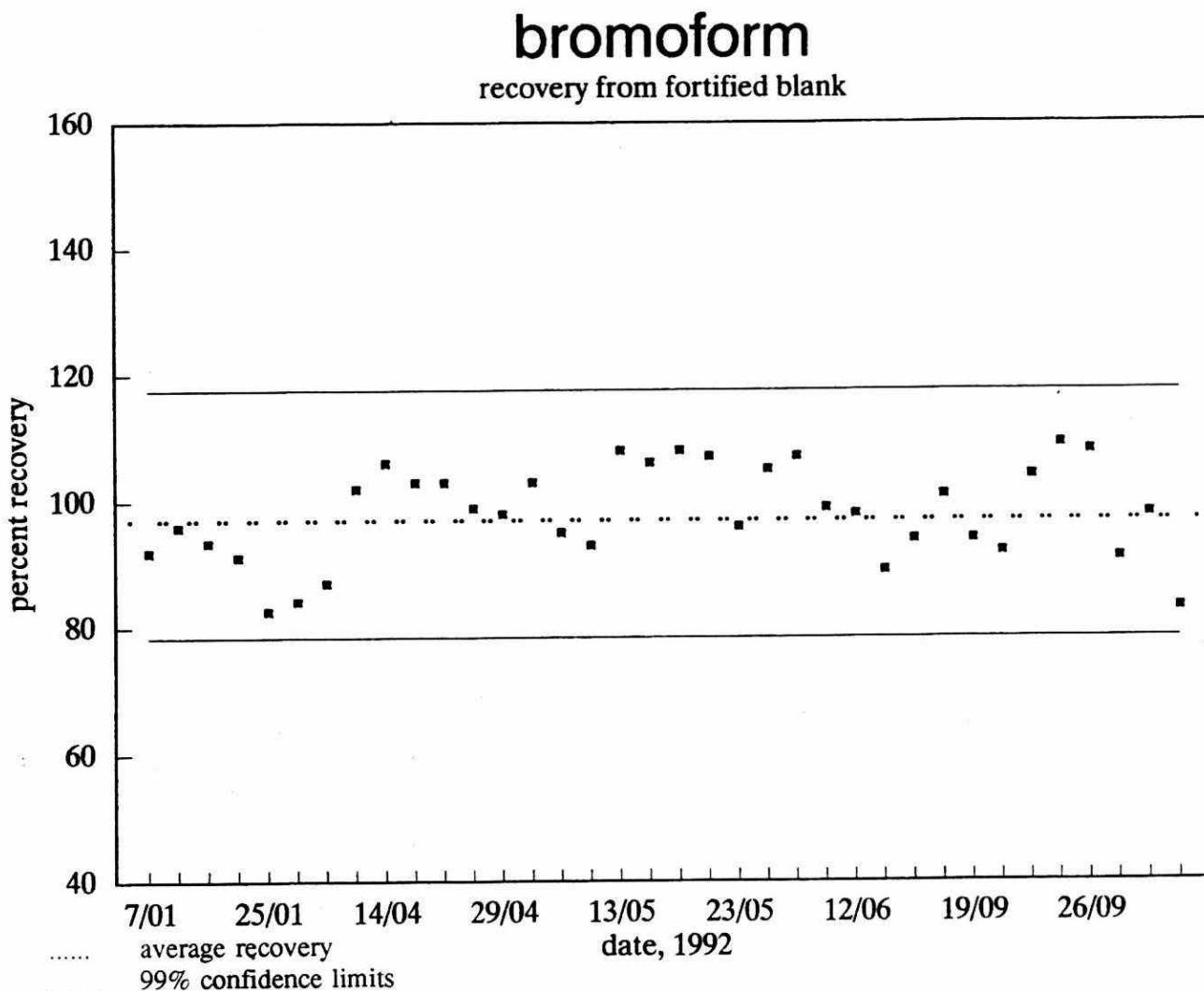
recovery from fortified blank



Performance Summary Table

January - December 1992

|                                    |                      |
|------------------------------------|----------------------|
| Analyte                            | dibromochloromethane |
| True Concentration                 | 20 µg/L              |
| Number of Observations             | 36                   |
| Within-run Rel. Standard Deviation | 3.8% (n=13)          |
| Between-run Standard Deviation     | 4.8%                 |
| Accuracy (% of expected)           | 97.3%                |



Performance Summary Table

January - December 1992

|                                    |           |
|------------------------------------|-----------|
| Analyte                            | bromoform |
| True Concentration                 | 10 µg/L   |
| Number of Observations             | 36        |
| Within-run Rel. Standard Deviation | 5% (n=13) |
| Between-run Standard Deviation     | 8%        |
| Accuracy (% of expected)           | 98%       |

**METHOD CODE :** OWOC-E3120B

**METHOD TITLE:** The Determination of Organochlorine Pesticides, Polychlorinated Biphenyls and Other Chlorinated Organic Compounds in Water by GC-ECD

**LABORATORY :** Organic Water Unit

**SUPERVISOR :** P. Crozier/ Dr. D. Hall

**SAMPLE TYPE :** surface water, groundwater, raw and treated drinking water

**PRINCIPLE OF THE METHOD :**

Samples are extracted with an organic solvent; the extract is dried, concentrated by rotary evaporator, cleaned-up on Florisil, and re-concentrated prior to analysis by dual column capillary gas chromatography with dual electron capture detection.

| <b>PARAMETERS MEASURED :</b>   | <b>LIS TEST CODE</b> | <b>W ( ng/L )</b> | <b>T ( ng/L )</b> |
|--------------------------------|----------------------|-------------------|-------------------|
| hexachloroethane               | X2HCE                | 1                 | 10                |
| 1,3,5-trichlorobenzene         | X2135                | 5                 | 50                |
| 1,2,4-trichlorobenzene         | X2124                | 5                 | 50                |
| 1,2,3-trichlorobenzene         | X2123                | 5                 | 50                |
| hexachlorobutadiene            | X1HCBD               | 1                 | 10                |
| 2,4,5-trichlorotoluene         | X2T245               | 5                 | 50                |
| 2,3,6-trichlorotoluene         | X2T236               | 5                 | 50                |
| 1,2,3,5-tetrachlorobenzene     | X21235               | 1                 | 10                |
| 1,2,4,5-tetrachlorobenzene     | X21245               | 1                 | 10                |
| 1,2,3,4-tetrachlorobenzene     | X21234               | 1                 | 10                |
| $\alpha$ ,2,6-trichlorotoluene | X2T26A               | 5                 | 50                |
| pentachlorobenzene             | X2PNCB               | 1                 | 10                |
| hexachlorobenzene              | X2HCB                | 1                 | 10                |
| heptachlor                     | P1HEPT               | 1                 | 10                |
| aldrin                         | P1ALDR               | 1                 | 10                |
| p,p'-DDE                       | P1PPDE               | 1                 | 10                |
| $\alpha$ -BHC                  | P1BHCA               | 1                 | 10                |
| $\beta$ -BHC                   | P1BHCB               | 1                 | 10                |
| $\gamma$ -BHC                  | P1BHCG               | 1                 | 10                |
| $\alpha$ -chlordane            | P1CHLA               | 2                 | 20                |
| $\gamma$ -chlordane            | P1CHLG               | 2                 | 20                |
| oxychlordane                   | P1OCHL               | 2                 | 20                |
| $\alpha$ , $\rho$ '-DDT        | P1OPDT               | 5                 | 50                |
| p,p'-DDD                       | P1PPDD               | 5                 | 50                |
| p,p'-DDT                       | P1PPDT               | 5                 | 50                |
| methoxychlor                   | P1DMDT               | 5                 | 50                |

( parameters measured continued )

|                           |        |     |       |
|---------------------------|--------|-----|-------|
| heptachlor epoxide        | P1HEPE | 1   | 10    |
| endosulfan I              | P1END1 | 2   | 20    |
| dieldrin                  | P1DIEL | 2   | 20    |
| endrin                    | P1ENDR | 5   | 50    |
| endosulfan II             | P1END2 | 5   | 50    |
| endosulfan cyclic sulfate | P1ENDS | 5   | 50    |
| mirex                     | P1MIRX | 5   | 50    |
| total PCB's               | P1PCBT | 20  | 200   |
| octachlorostyrene         | X2OCST | 1   | 10    |
| toxaphene                 | P1TOX  | 500 | 5 000 |

#### REPORTING FORMAT :

Results are reported in parts per trillion ( ng/L ) rounded off to the closest increment of W and up to maximum of two significant figures.

#### QUALITY CONTROL :

The routine quality control operations monitor validity of calibration ( calibration check solution ), required instrument sensitivity ( low level check solution ), absence of potential interferences ( method blanks ) and overall method performance ( fortified method blanks ).

For selected target compounds, control charts summarizing the response factors used to calibrate instruments and the recoveries from fortified method blanks are maintained.

**REMARKS :** In addition to the intra-laboratory method control, the performance of the method was examined through performance audit samples program organized by LSB Quality Management Office.

In 1992 the method participated in the Smithville Tender Intercomparison Study organized by the Laboratory Services Branch.

List of Performance Charts :      Hexachlorobenzene ( recovery from fortified blank )  
                                        1,3,5-Trichlorobenzene ( recovery from fortified blank )  
                                        Hexachlorobutadiene ( recovery from fortified blank )  
                                        Mirex ( recovery from fortified blank )  
                                        Total PCB ( recovery from fortified blank )

List of Performance Tables :      Method Blanks Summary  
                                        Hexachlorobenzene  
                                        1,3,5-Trichlorobenzene  
                                        Hexachlorobutadiene  
                                        Mirex  
                                        Total PCB

Method Blanks Summary

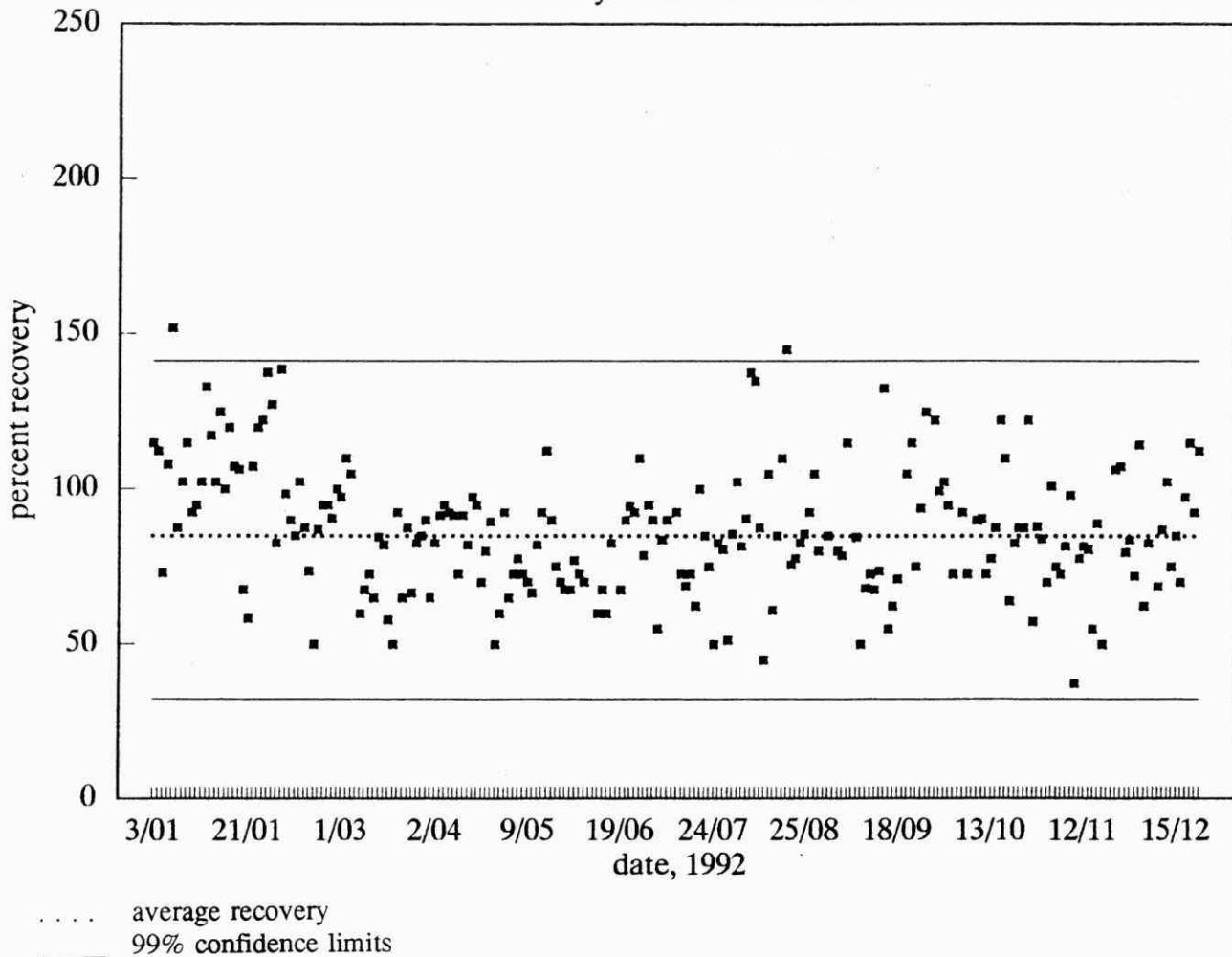
January 1992 - December 1992

| Analyte                        | Number of Observations | Average Concentration ( ng/L ) | Standard Deviation ( ng/L ) |
|--------------------------------|------------------------|--------------------------------|-----------------------------|
| hexachloroethane               | 126                    | ND ( 0.1 )                     |                             |
| 1,3,5-trichlorobenzene         | 126                    | ND ( 2 )                       |                             |
| 1,2,4-trichlorobenzene         | 126                    | 0.4                            | 4.6                         |
| 1,2,3-trichlorobenzene         | 126                    | ND ( 2 )                       |                             |
| hexachlorobutadiene            | 126                    | 0.1                            | 1.3                         |
| 2,4,5-trichlorotoluene         | 126                    | ND ( 3 )                       |                             |
| 2,3,6-trichlorotoluene         | 126                    | ND ( 3 )                       |                             |
| 1,2,3,5-tetrachlorobenzene     | 126                    | ND ( 1 )                       |                             |
| 1,2,4,5-tetrachlorobenzene     | 126                    | ND ( 2 )                       |                             |
| 1,2,3,4-tetrachlorobenzene     | 126                    | ND ( 0.9 )                     |                             |
| $\alpha$ ,2,6-trichlorotoluene | 126                    | ND ( 0.7 )                     |                             |
| pentachlorobenzene             | 126                    | 0.1                            | 2.0                         |
| hexachlorobenzene              | 126                    | ND ( 0.5 )                     |                             |
| heptachlor                     | 126                    | ND ( 0.5 )                     |                             |
| aldrin                         | 126                    | ND ( 0.5 )                     |                             |
| p,p'-DDE                       | 126                    | ND ( 0.6 )                     |                             |
| $\alpha$ -BHC                  | 126                    | ND ( 0.5 )                     |                             |
| $\beta$ -BHC                   | 126                    | ND ( 1 )                       |                             |
| $\gamma$ -BHC                  | 126                    | ND ( 0.4 )                     |                             |
| $\alpha$ -chlordane            | 126                    | ND ( 0.6 )                     |                             |
| $\gamma$ -chlordane            | 126                    | ND ( 0.6 )                     |                             |
| oxychlordane                   | 126                    | ND ( 0.7 )                     |                             |
| $\alpha$ ,p'-DDT               | 126                    | ND ( 1 )                       |                             |
| p,p'-DDD                       | 126                    | ND ( 0.8 )                     |                             |
| p,p'-DDT                       | 126                    | ND ( 0.8 )                     |                             |
| methoxychlor                   | 126                    | ND ( 1 )                       |                             |
| heptachlor epoxide             | 126                    | ND ( 0.5 )                     |                             |
| endosulfan I                   | 126                    | ND ( 0.5 )                     |                             |
| dieldrin                       | 126                    | ND ( 0.6 )                     |                             |
| endosulfan II                  | 126                    | ND ( 0.7 )                     |                             |
| endosulfan cyclic sulfate      | 126                    | ND ( 0.7 )                     |                             |
| mirex                          | 126                    | 0.03                           | 0.21                        |
| total PCB's                    | 126                    | 0.9                            | 4.7                         |
| octachlorostyrene              | 126                    | ND ( 0.5 )                     |                             |

ND ... Not detected. Detection limit in ng/L given in brackets ( ).

## hexachlorobenzene

recovery from fortified blank



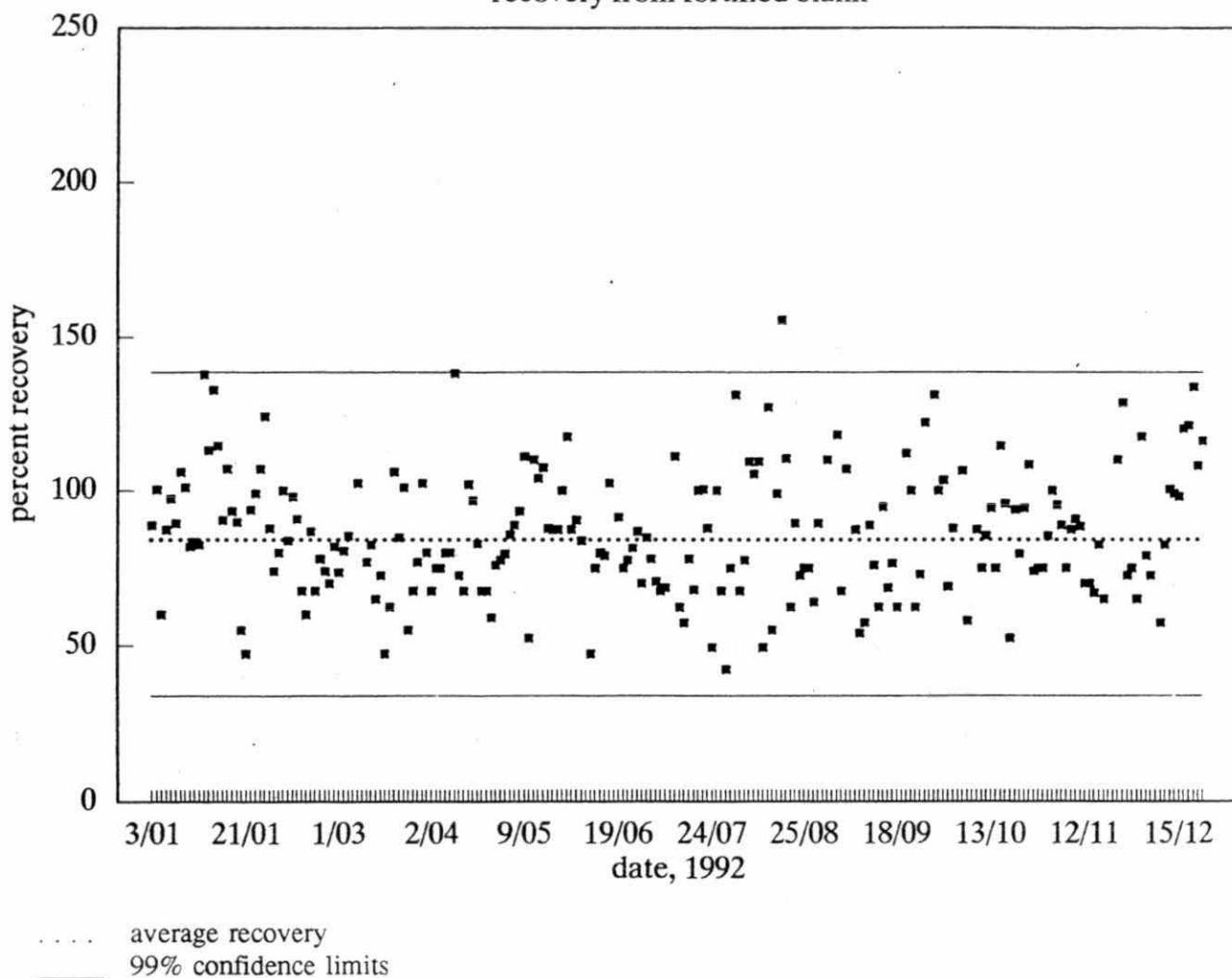
### Method Performance Summary

January - December 1992

|                                |                   |
|--------------------------------|-------------------|
| Analyte                        | hexachlorobenzene |
| True Concentration             | 100 ng/L          |
| Number of Observations         | 217               |
| Between-run Standard Deviation | 23%               |
| Accuracy (% of expected)       | 85%               |

## 1,3,5-trichlorobenzene

recovery from fortified blank



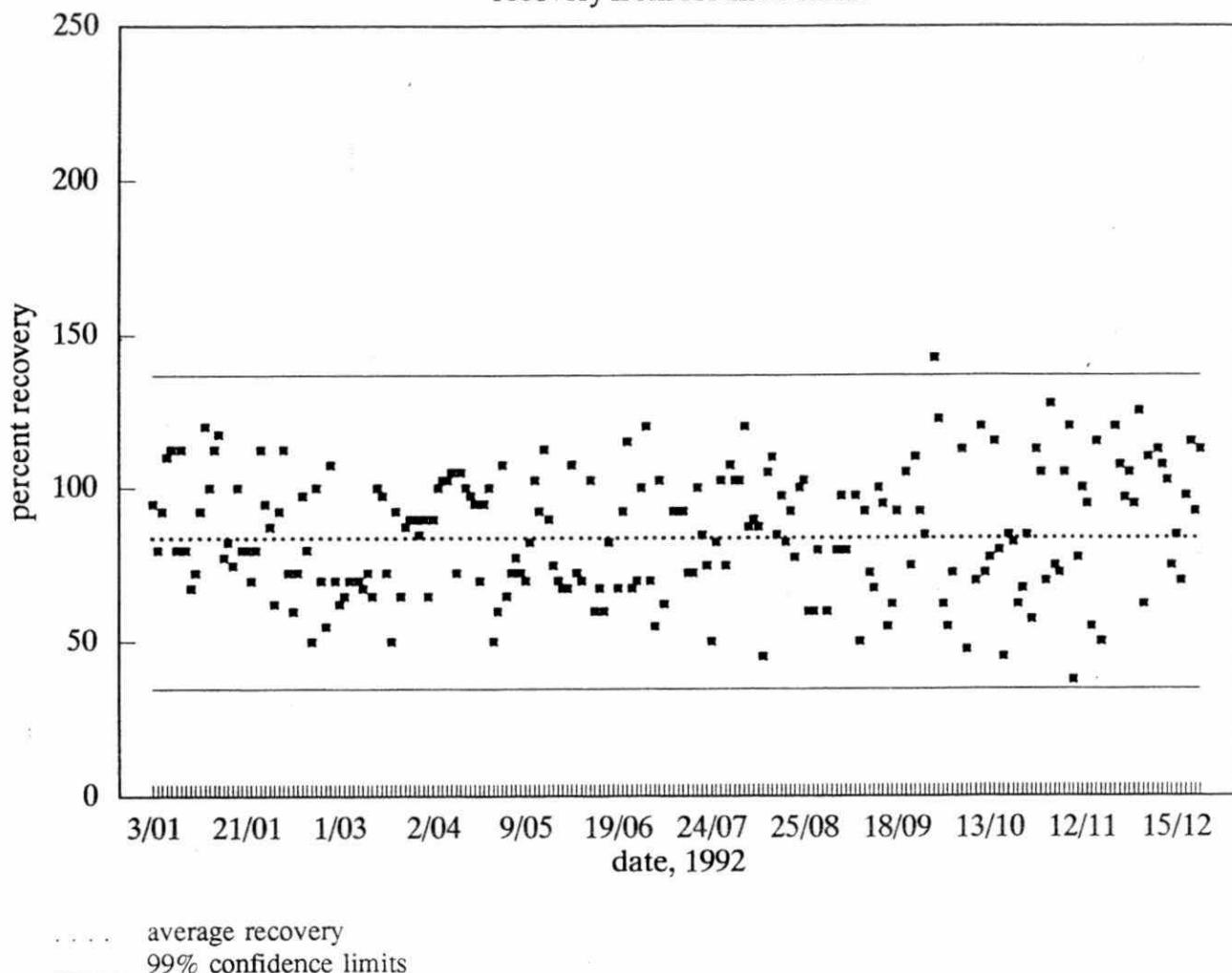
### Method Performance Summary

January - December 1992

|                                |                        |
|--------------------------------|------------------------|
| Analyte                        | 1,3,5-trichlorobenzene |
| True Concentration             | 100 ng/L               |
| Number of Observations         | 212                    |
| Between-run Standard Deviation | 21%                    |
| Accuracy (% of expected)       | 86%                    |

## hexachlorobutadiene

recovery from fortified blank



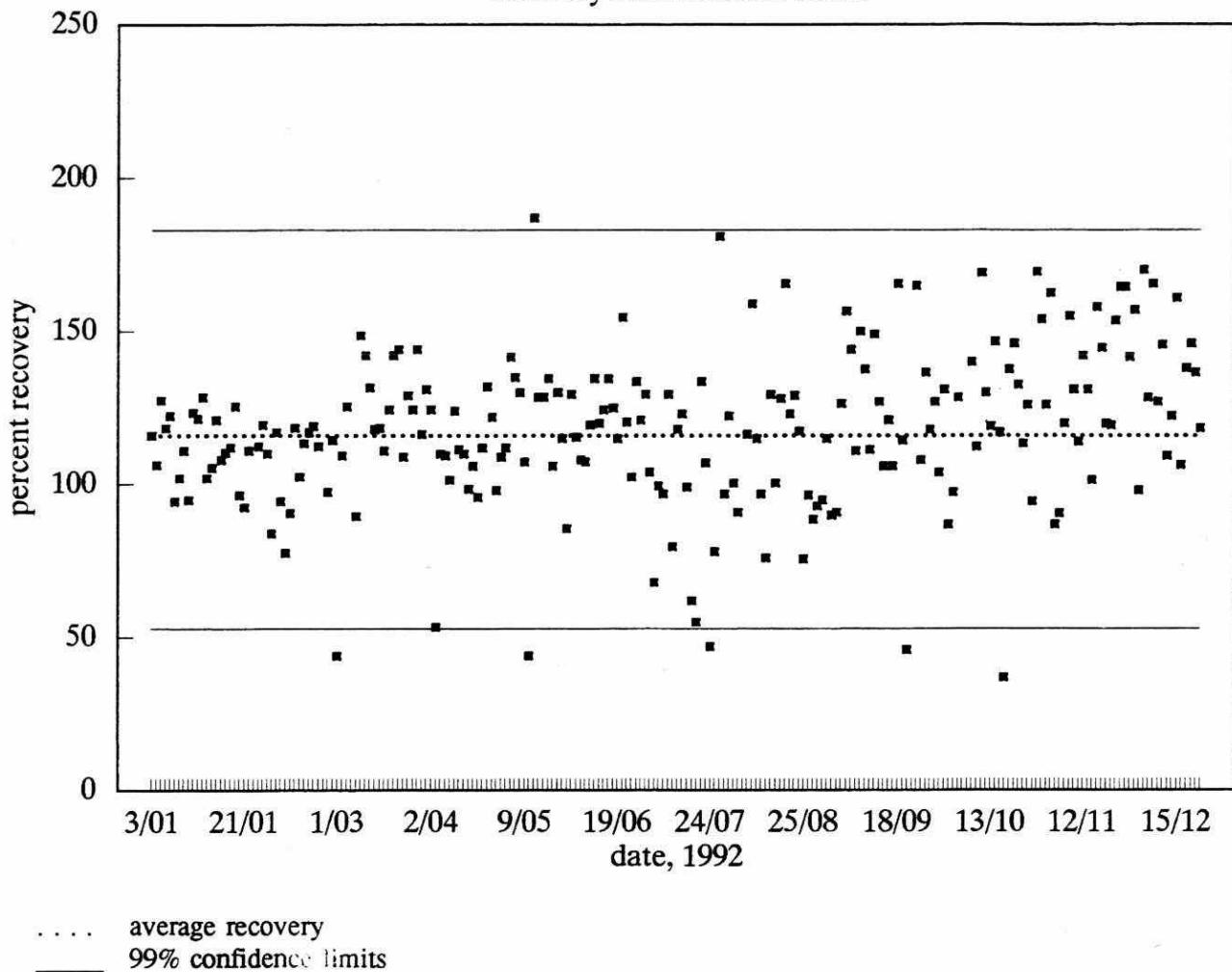
### Method Performance Summary

January - December 1992

|                                |                     |
|--------------------------------|---------------------|
| Analyte                        | hexachlorobutadiene |
| True Concentration             | 20 ng/L             |
| Number of Observations         | 216                 |
| Between-run Standard Deviation | 21%                 |
| Accuracy (% of expected)       | 85%                 |

# mirex

recovery from fortified blank



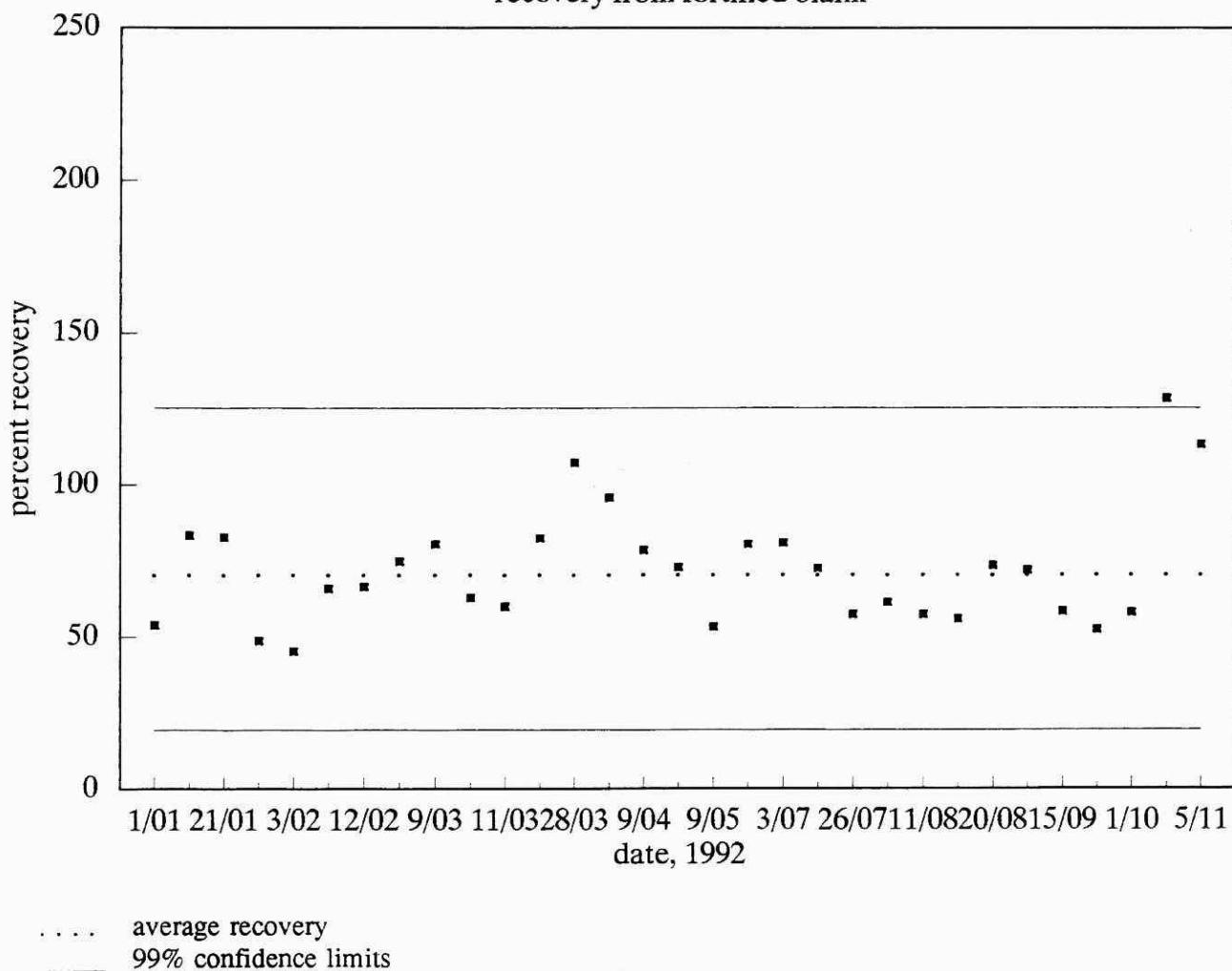
## Method Performance Summary

January - December 1992

| Analyte                        | mirex    |
|--------------------------------|----------|
| True Concentration             | 100 ng/L |
| Number of Observations         | 219      |
| Between-run Standard Deviation | 25%      |
| Accuracy (% of expected)       | 118%     |

## total PCB ( Aroclor 1254/ Aroclor 1260 )

recovery from fortified blank



### Method Performance Summary

January - December 1992

|                                |  |
|--------------------------------|--|
| Analyte                        | total PCB ( aroclor 1254/ aroclor 1260 ) |
| True Concentration             | 200 ng/L                                 |
| Number of Observations         | 31                                       |
| Between-run Standard Deviation | 19%                                      |
| Accuracy (% of expected)       | 72%                                      |

**METHOD CODE :** OWCP-B-E3119A

**METHOD TITLE:** The Determination of Chlorophenols and Phenoxyacid Herbicides in Water by Solid Phase Extraction (SPE) and GC-ECD

**LABORATORY :** Organic Water Unit

**SUPERVISOR :** P. Crozier / Dr. D. Hall

**SAMPLE TYPE :** surface water, groundwater, raw and treated drinking water

**PRINCIPLE OF THE METHOD :**

The aqueous sample is aspirated through C-18 bonded porous silica cartridge. The cartridge is eluted with a small volume of solvent. The eluate is methylated and the 2,4-D type herbicides and chlorophenols are determined as the corresponding methyl esters and ethers by dual capillary gas chromatography with electron capture detection.

| <b>PARAMETERS MEASURED :</b>      | <b>LIS TEST CODE</b> | <b>W ( ng/L )</b> | <b>T ( ng/L )</b> |
|-----------------------------------|----------------------|-------------------|-------------------|
| 2,4,6-trichlorophenol             | X3246                | 20                | 200               |
| 2,4,5-trichlorophenol             | X3245                | 100               | 1 000             |
| 2,3,4-trichlorophenol             | X3234                | 100               | 1 000             |
| 2,3,5,6-tetrachlorophenol         | X32356               | 10                | 100               |
| 2,3,4,5-tetrachlorophenol         | X32345               | 20                | 200               |
| pentachlorophenol                 | X3PCPH               | 10                | 100               |
| Dicamba                           | P3DICA               | 50                | 500               |
| 2,4-dichlorophenoxypropanoic acid | P324DP               | 100               | 1 000             |
| 2,4-dichlorophenoxyacetic acid    | P324D                | 100               | 1 000             |
| Silvex                            | P3SILV               | 20                | 200               |
| 2,4,5-trichlorophenoxyacetic acid | P3245T               | 50                | 500               |
| 2,4-dichlorophenoxybutyric acid   | P324DB               | 200               | 2 000             |

**REPORTING FORMAT :**

Results are reported in parts per trillion (ng/L) rounded off to the closest increment of W and up to maximum of two significant figures.

**QUALITY CONTROL :**

Quality control samples included in the run format are method blanks, fortified method blanks and calibration check solutions.

For selected target compounds, control charts summarizing the response factors used to calibrate instruments and the recoveries from fortified method blanks are maintained.

**REMARKS :** In addition to the intra-laboratory method control, the performance of the method was

examined through performance audit samples program organized by LSB Quality Management Office.

List of Performance Charts :      2,4,6-Trichlorophenol ( recovery from fortified blank )  
    2,4-Dichlorophenoxyacetic Acid ( recovery from fortified blank )  
    Silvex ( recovery from fortified blank )

List of Performance Tables :      Method Blanks Summary  
    2,4,6-Trichlorophenol  
    2,4-Dichlorophenoxyacetic Acid  
    Silvex

Method Blanks Summary

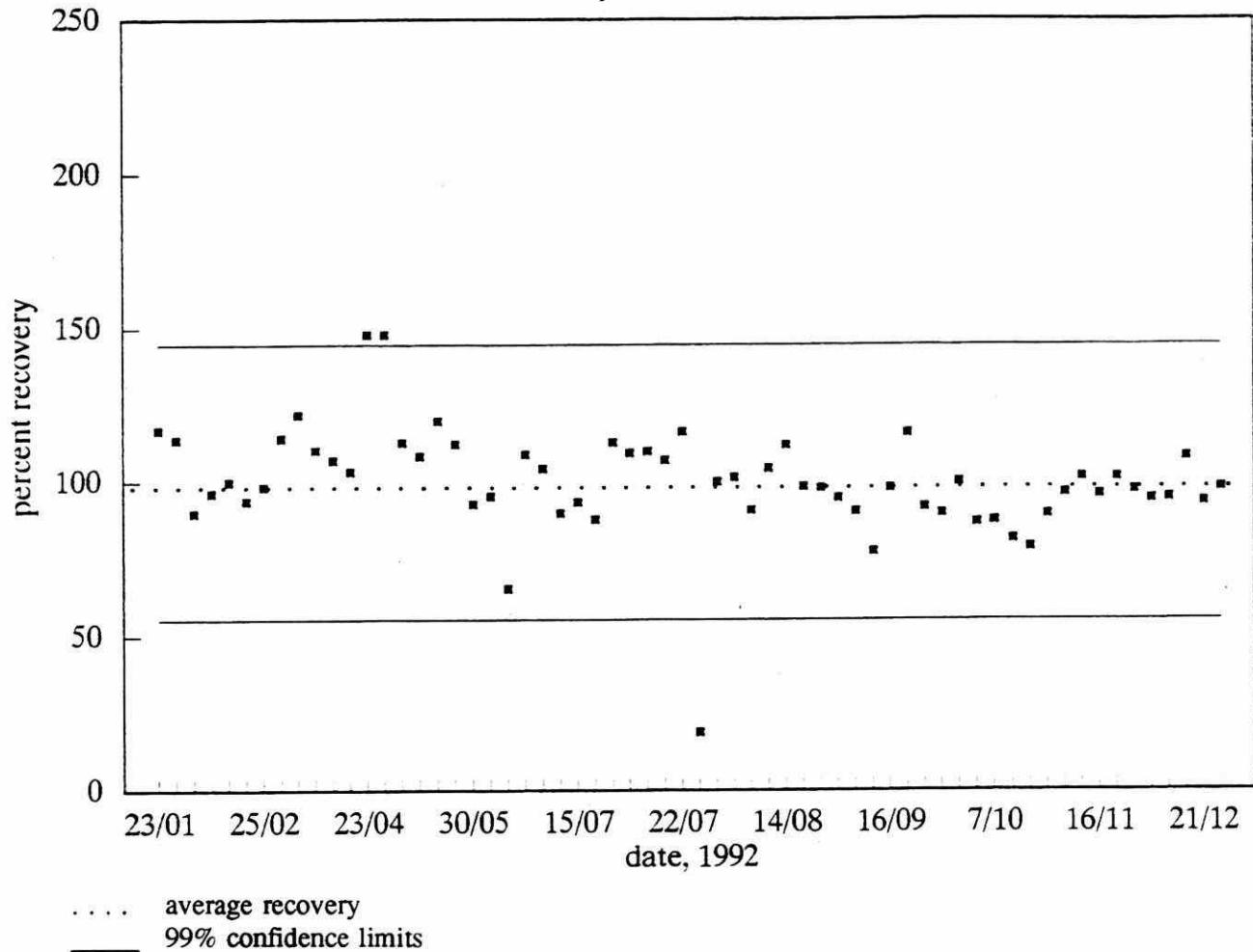
January 1992 - December 1992

| Analyte                           | Number of Observations | Average Concentration ( ng/L ) | Standard Deviation ( ng/L ) |
|-----------------------------------|------------------------|--------------------------------|-----------------------------|
| 2,4,6-trichlorophenol             | 63                     | ND ( 2 )                       |                             |
| 2,4,5-trichlorophenol             | 63                     | ND ( 3 )                       |                             |
| 2,3,4-trichlorophenol             | 63                     | ND ( 2 )                       |                             |
| 2,3,5,6-tetrachlorophenol         | 63                     | ND ( 1 )                       |                             |
| 2,3,4,5-tetrachlorophenol         | 63                     | 0.7                            | 1.7                         |
| pentachlorophenol                 | 63                     | 5                              | 13                          |
| Dicamba                           | 63                     | ND ( 3 )                       |                             |
| 2,4-dichlorophenoxypropanoic acid | 63                     | ND ( 10 )                      |                             |
| 2,4-dichlorophenoxyacetic acid    | 63                     | ND ( 10 )                      |                             |
| Silvex                            | 63                     | ND ( 2 )                       |                             |
| 2,4,5-trichlorophenoxyacetic acid | 63                     | ND ( 2 )                       |                             |
| 2,4-dichlorophenoxybutyric acid   | 63                     | ND ( 20 )                      |                             |
| Picloram                          | 63                     | ND ( 2 )                       |                             |

ND ... Not detected. Detection limit in ng/L given in brackets ( ).

## 2,4,6-trichlorophenol

recovery from fortified blank



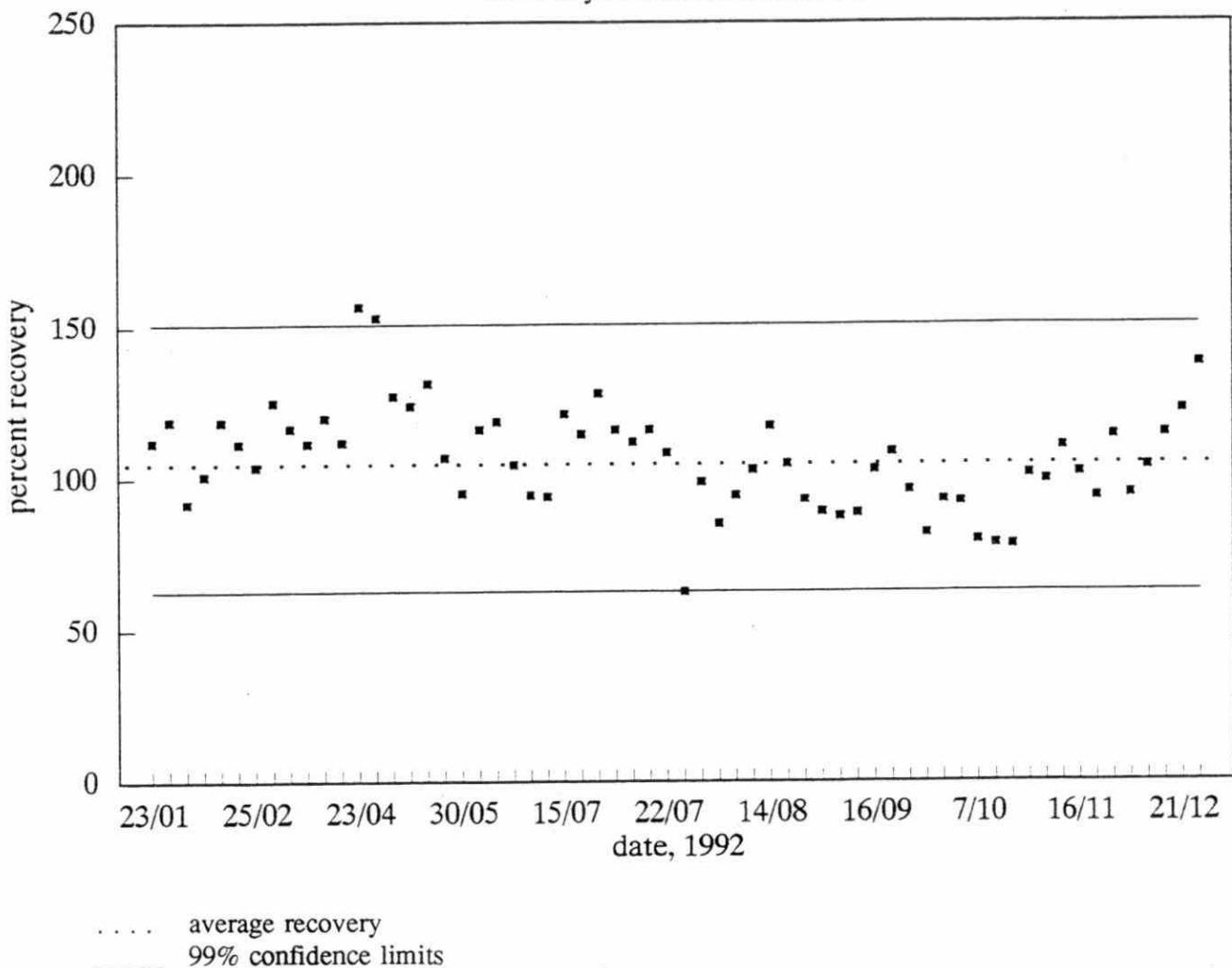
### Method Performance Summary

January - December 1992

|                                |                       |
|--------------------------------|-----------------------|
| Analyte                        | 2,4,6-trichlorophenol |
| True Concentration             | 100 ng/L              |
| Number of Observations         | 62                    |
| Between-run Standard Deviation | 17%                   |
| Accuracy (% of expected)       | 100%                  |

## 2,4-dichlorophenoxyacetic acid

recovery from fortified blank



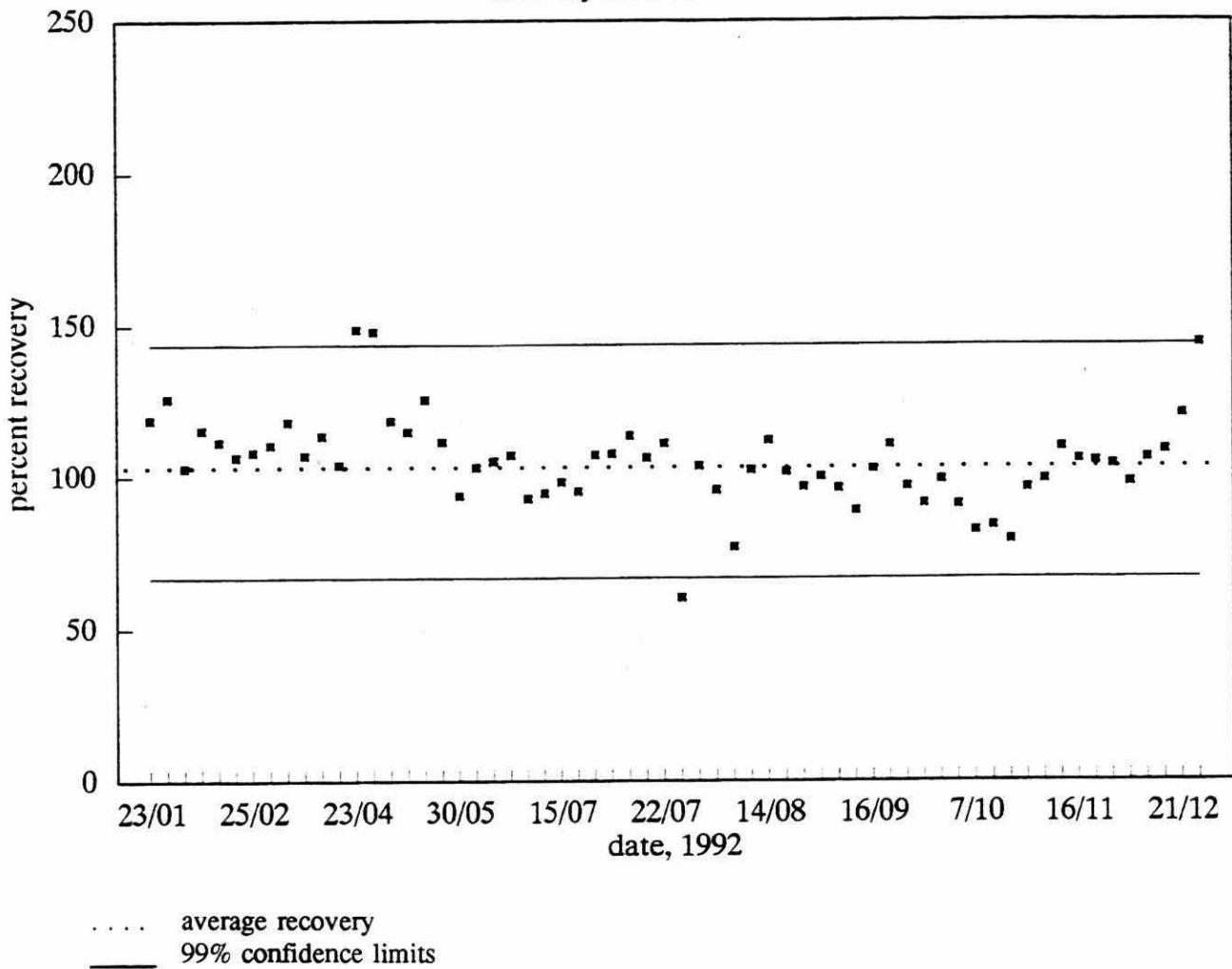
### Method Performance Summary

January - December 1992

|                                |                                |
|--------------------------------|--------------------------------|
| Analyte                        | 2,4-dichlorophenoxyacetic acid |
| True Concentration             | 750 ng/L                       |
| Number of Observations         | 62                             |
| Between-run Standard Deviation | 17%                            |
| Accuracy (% of expected)       | 107%                           |

# silvex

recovery from fortified blank



### Method Performance Summary

January - December 1992

|                                |          |
|--------------------------------|----------|
| Analyte                        | silvex   |
| True Concentration             | 150 ng/L |
| Number of Observations         | 62       |
| Between-run Standard Deviation | 14%      |
| Accuracy (% of expected)       | 105%     |

**METHOD CODE :** OWTRI-E3121A

**METHOD TITLE:** The Determination of Triazine Herbicides in Water by GC-TSD

**LABORATORY :** Organic Water

**SUPERVISOR :** P. Crozier / Dr. D. Hall

**SAMPLE TYPE :** surface water, groundwater, raw and treated drinking water

**PRINCIPLE OF THE METHOD :**

Sample pH is adjusted to 12 and the triazine herbicides are extracted with an organic solvent. The extract is dried and then evaporated to dryness. The reconstituted extract is examined by gas chromatography using a thermionic specific detector.

| <b>PARAMETERS MEASURED :</b> | <b>LIS TEST CODE</b> | <b>W ( ng/L )</b> | <b>T ( ng/L )</b> |
|------------------------------|----------------------|-------------------|-------------------|
| prometon                     | P2PROM               | 50                | 500               |
| atraton                      | P2ATRO               | 50                | 500               |
| propazine                    | P2PROP               | 50                | 500               |
| atrazine                     | P2ATRA               | 50                | 500               |
| prometryne                   | P2PROY               | 50                | 500               |
| simazine                     | P2SIM                | 50                | 500               |
| ametryne                     | P2AMET               | 50                | 500               |
| sencor                       | P2SENC               | 100               | 1 000             |
| bladex                       | P2BLAD               | 100               | 1 000             |
| metolachlor                  | P0MET                | 500               | 5 000             |
| alachlor                     | P0LASS               | 500               | 5 000             |
| desethyl atrazine            | P2DATR               | 200               | 2 000             |
| desethyl simazine            | P2DSIM               | 200               | 2 000             |

**REPORTING FORMAT :**

Results are reported in parts per trillion (ng/L) rounded off to the closest increment of W up to maximum of two significant figures.

**QUALITY CONTROL :**

The routine quality control operations monitor validity of calibration ( calibration check solution ), maintenance of required instrument sensitivity ( low level check solution ), absence of potential interferences ( method blanks ) and overall method performance ( fortified method blanks ).

Control charts summarizing the response factors used to calibrate instruments and the recoveries from fortified method blanks are maintained for selected target compounds.

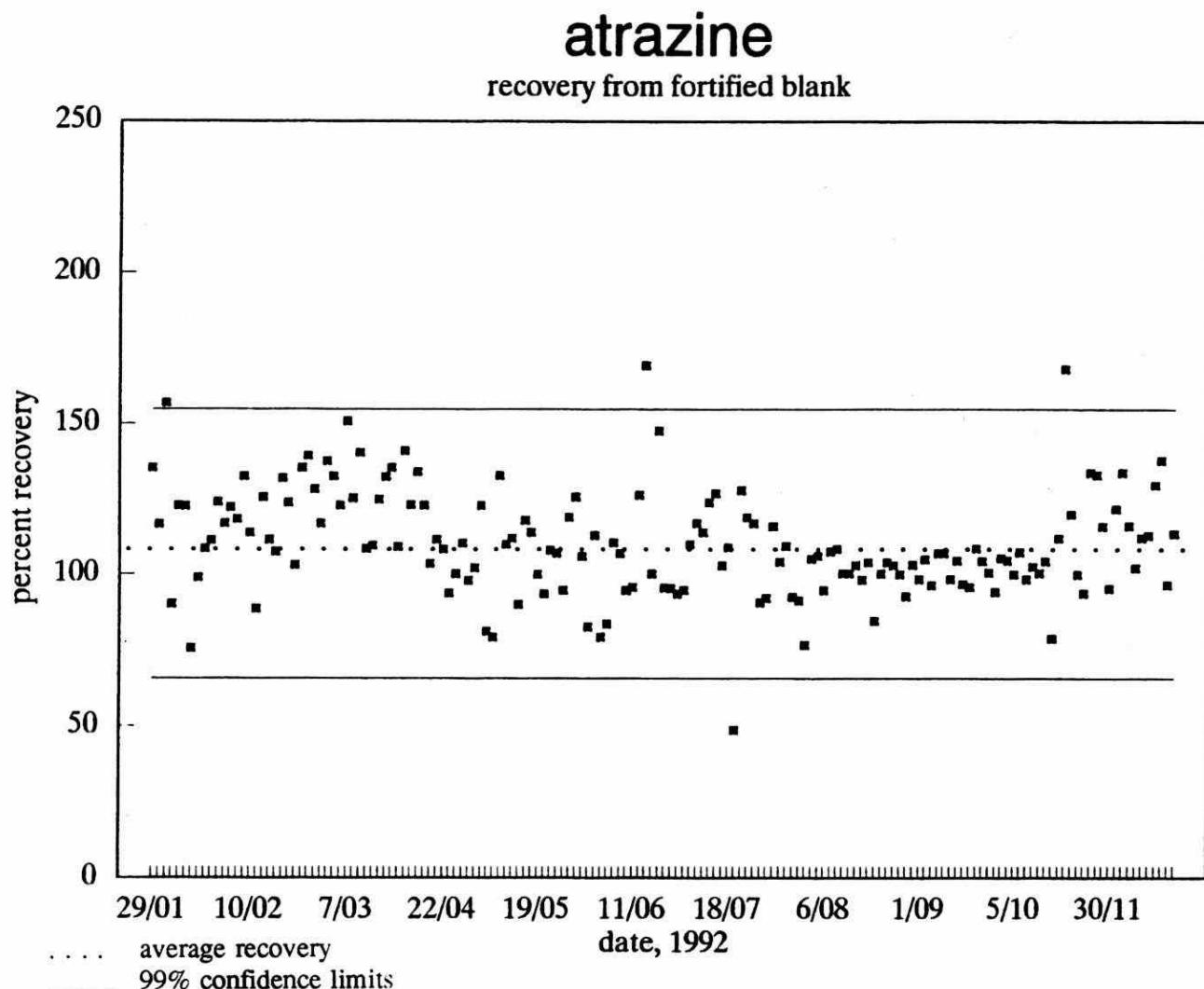
**REMARKS :** During the period starting January 1992 and ending December 1992, a total of 172 procedure blanks were prepared and tested by the method. For these 172 analyses, no observable responses of any of the target analytes were encountered.

In addition to the intra-laboratory method control, the performance of the method was examined through performance audit samples program organized by LSB Quality Management Office.

In July 1992, the performance of the method was examined through the CAPCO Interlaboratory QC Study organized by the National Water Research Institute.

List of Performance Charts and Tables:

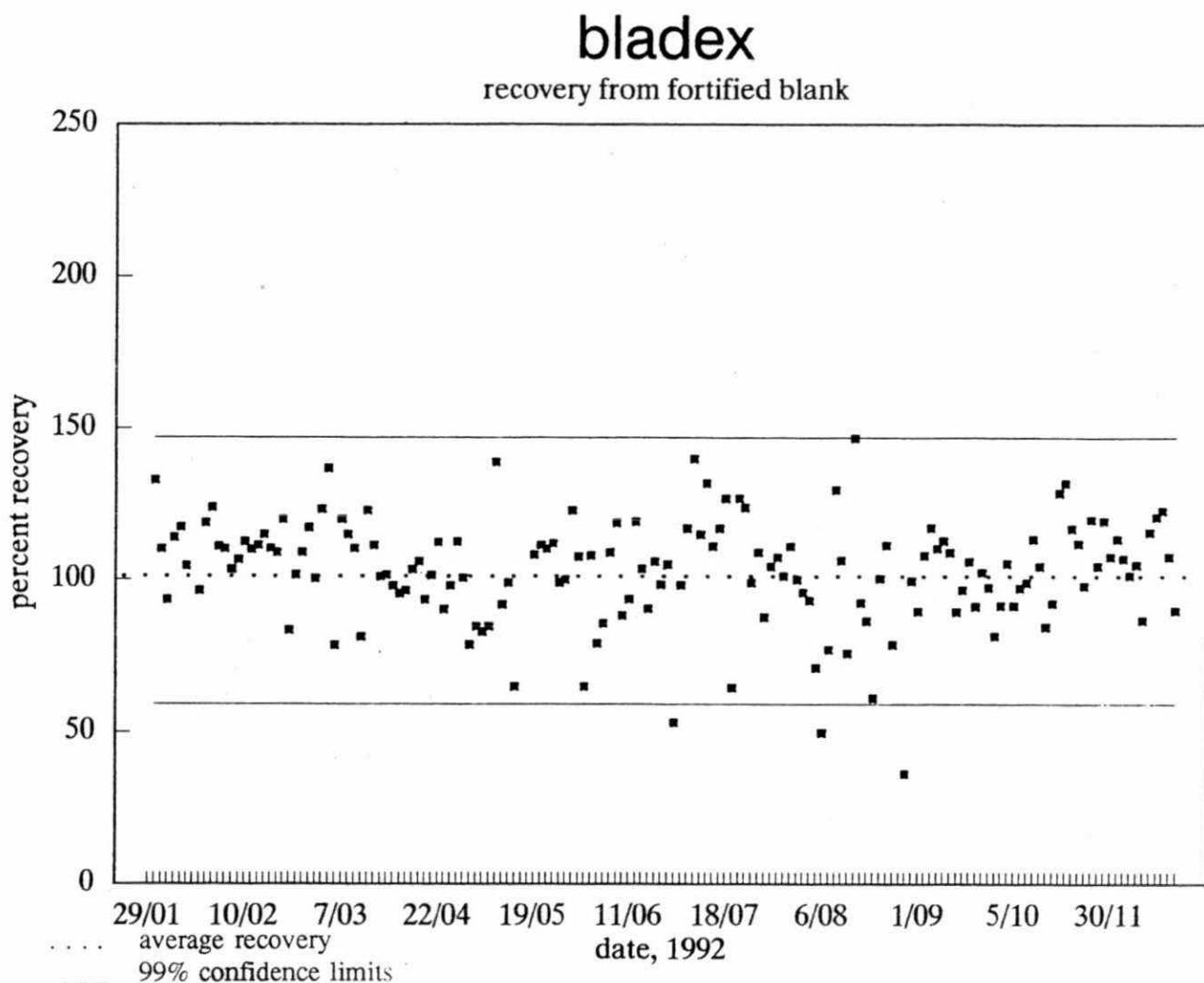
Atrazine ( recovery from fortified blank )  
Bladex ( recovery from fortified blank )  
Metolachlor ( recovery from fortified blank )



**Method Performance Summary**

January - December 1992

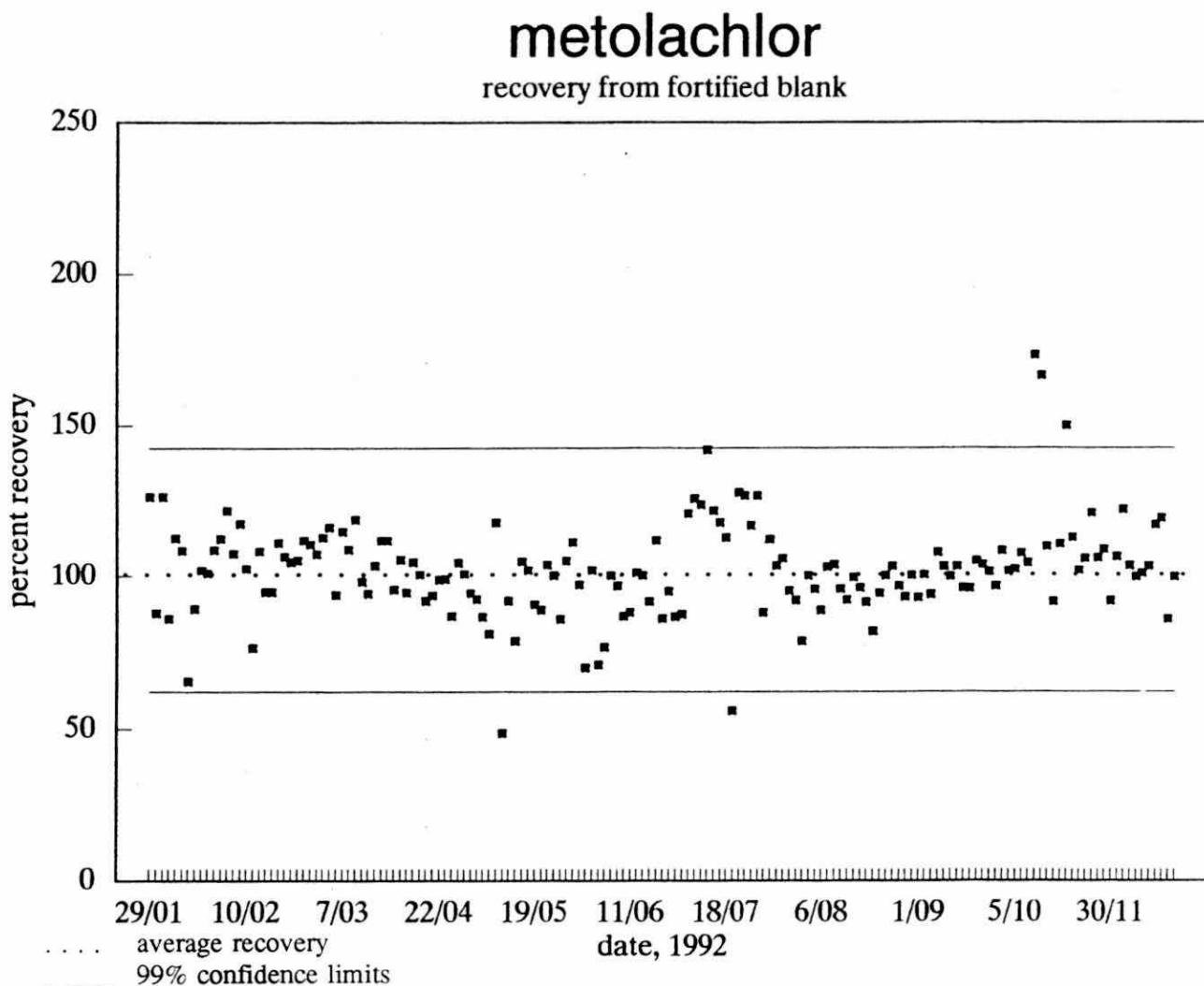
|                                |          |
|--------------------------------|----------|
| Analyte                        | atrazine |
| True Concentration             | 200 ng/L |
| Number of Observations         | 161      |
| Between-run Standard Deviation | 18%      |
| Accuracy (% of expected)       | 110%     |



**Method Performance Summary**

January - December 1992

|                                |          |
|--------------------------------|----------|
| Analyte                        | bladex   |
| True Concentration             | 200 ng/L |
| Number of Observations         | 156      |
| Between-run Standard Deviation | 17%      |
| Accuracy (% of expected)       | 103%     |



Method Performance Summary

January - December 1992

|                                |             |
|--------------------------------|-------------|
| Analyte                        | metolachlor |
| True Concentration             | 1 000 ng/L  |
| Number of Observations         | 161         |
| Between-run Standard Deviation | 16%         |
| Accuracy (% of expected)       | 102%        |

**METHOD CODE :** PWAOP-E32224A

**METHOD TITLE:** The Determination of Organophosphorus Pesticides in Water by GC-TSD

**LABORATORY :** Organic Water Unit

**SUPERVISOR :** P. Crozier / Dr. D. Hall

**SAMPLE TYPE :** surface water, groundwater, raw and treated drinking water

**PRINCIPLE OF THE METHOD :**

Samples are extracted with an organic solvent; water is removed from extract and extract is evaporated to dryness. The reconstituted extract is examined by dual capillary gas chromatography with a thermionic specific detector.

| <b>PARAMETERS MEASURED :</b> | <b>LIS TEST CODE</b> | <b>W ( ng/L )</b> | <b>T ( ng/L )</b> |
|------------------------------|----------------------|-------------------|-------------------|
| methyl trithion              | P4MTRI               | 20                | 200               |
| dichlorvos                   | P4DICH               | 20                | 200               |
| mevinphos                    | P4MEVI               | 20                | 200               |
| phorate ( thimet )           | P4PHOR               | 20                | 200               |
| diazinon                     | P4DIAZ               | 20                | 200               |
| ronnel                       | P4RONN               | 20                | 200               |
| chlorpyriphos ( dursban )    | P4DURS               | 20                | 200               |
| reldan                       | P4RELD               | 20                | 200               |
| malathion                    | P4MALA               | 20                | 200               |
| parathion                    | P4PARA               | 20                | 200               |
| methyl parathion             | P4MPAR               | 50                | 500               |
| ethion                       | P4ETHI               | 20                | 200               |

**REPORTING FORMAT :**

Results are reported in parts per trillion (ng/L) rounded off to the closest increment of W and up to maximum of two significant figures.

**QUALITY CONTROL :**

The routine quality control operations monitor validity of calibration ( calibration check solution ), absence of potential interferences ( method blanks ), overall method performance ( fortified method blanks ).

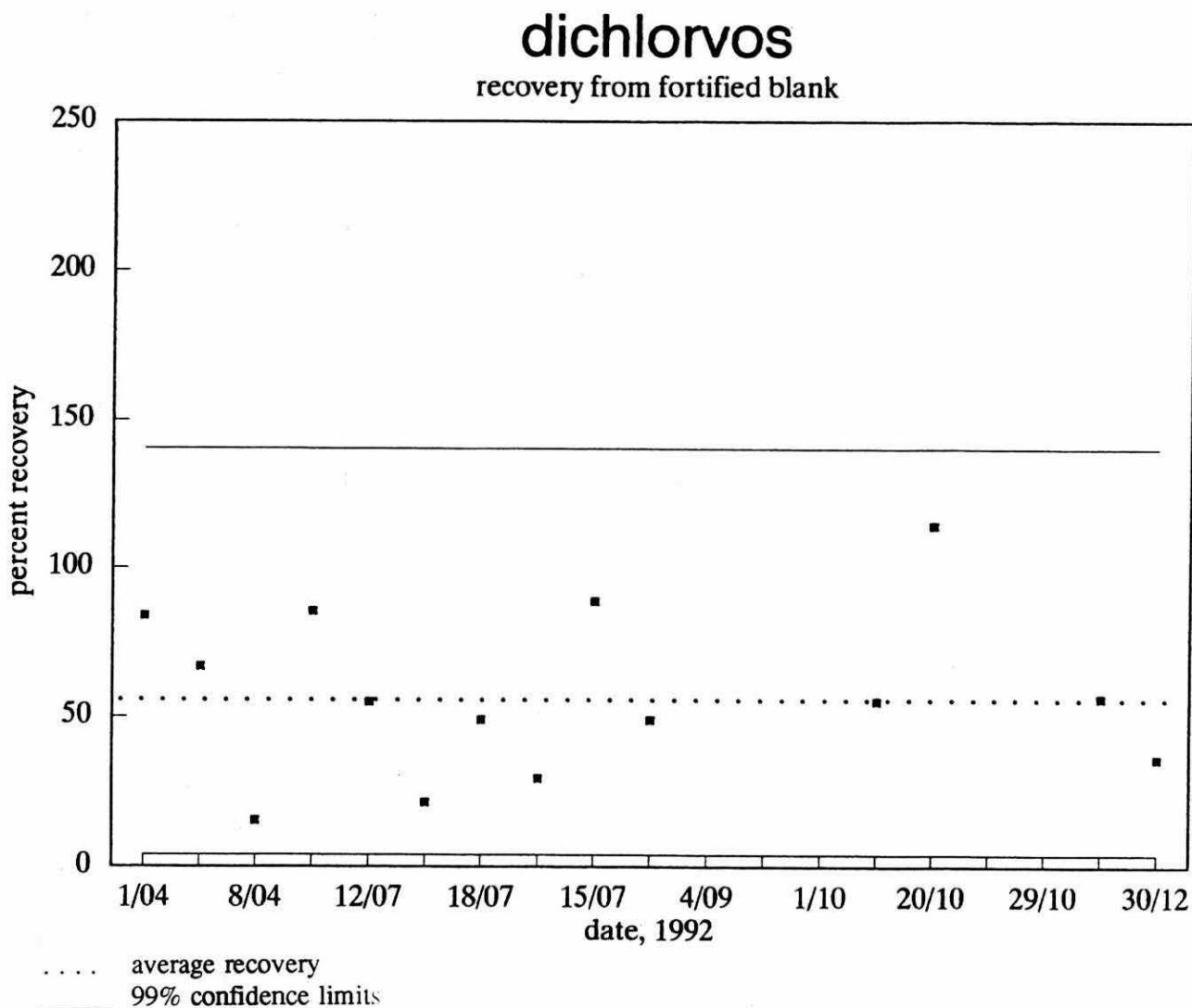
For selected target compounds, control charts summarizing the response factors used to calibrate instruments and the recoveries from fortified method blanks are maintained.

**REMARKS :** During the period starting January 1992 and ending December 1992, a total of 25 method blanks were prepared and tested by the method. For these 25 analyses, no observable responses of any of the target analytes were encountered.

In addition to the intra-laboratory method control, the performance of the method was examined through performance audit samples program organized by LSB Quality Management Office.

List of Performance Charts : Dichlorvos ( recovery from fortified blank )  
Diazinon ( recovery from fortified blank )  
Ethion ( recovery from fortified blank )

List of Performance Tables : **Dichlorvos**  
**Diazinon**  
**Ethion**



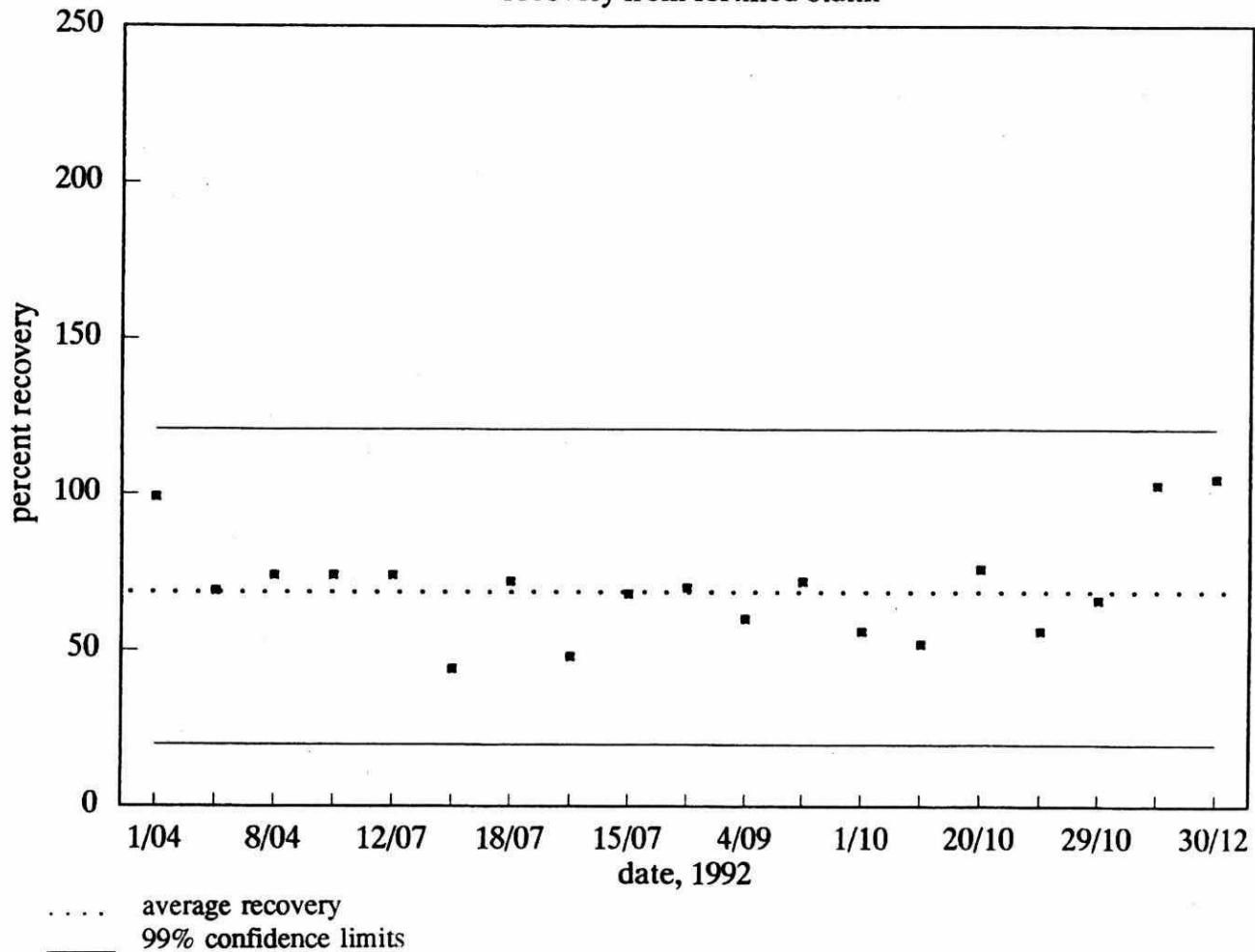
Method Performance Summary

January - December 1992

|                                |            |
|--------------------------------|------------|
| Analyte                        | dichlorvos |
| True Concentration             | 100 ng/L   |
| Number of Observations         | 14         |
| Between-run Standard Deviation | 27%        |
| Accuracy (% of expected)       | 58%        |

## diazinon

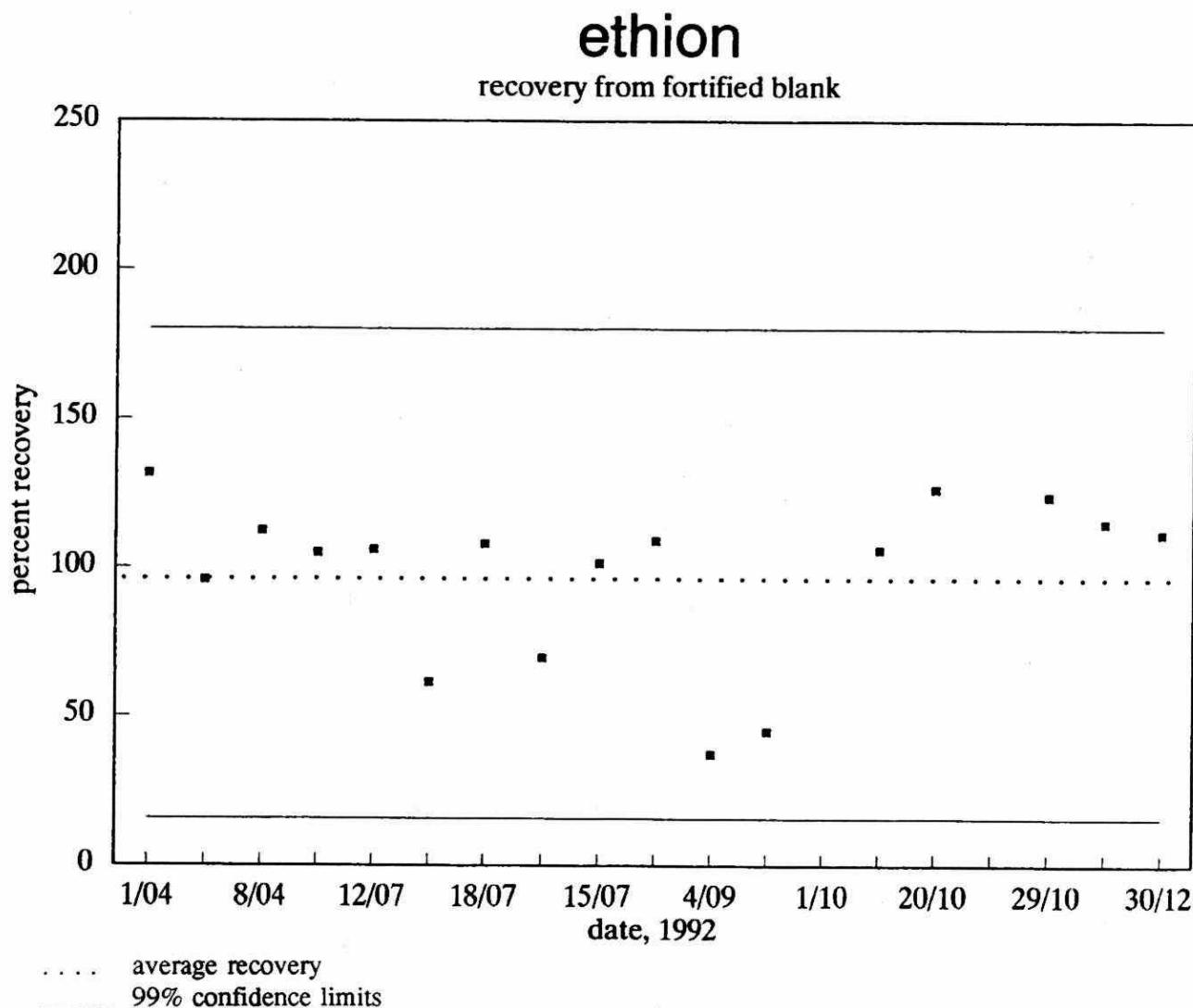
recovery from fortified blank



### Method Performance Summary

January - December 1992

|                                |          |
|--------------------------------|----------|
| Analyte                        | diazinon |
| True Concentration             | 100 ng/L |
| Number of Observations         | 19       |
| Between-run Standard Deviation | 17%      |
| Accuracy (% of expected)       | 70%      |



**Method Performance Summary**

January - December 1992

|                                |          |
|--------------------------------|----------|
| Analyte                        | ethion   |
| True Concentration             | 200 ng/L |
| Number of Observations         | 17       |
| Between-run Standard Deviation | 36%      |
| Accuracy (% of expected)       | 89%      |

**METHOD CODE :** HPLC/L-E3086A

**METHOD TITLE:** The Determination of Polynuclear Aromatic Hydrocarbons (PAHs) in Water by HPLC - Fluorescence Detection

**LABORATORY :** Organic Water Unit

**SUPERVISOR :** P. Crozier / Dr. D. Hall

**SAMPLE TYPE :** surface water, groundwater, raw and treated drinking water

**PRINCIPLE OF THE METHOD :**

Sample is extracted with an organic solvent; the extract is dried and evaporated to dryness. The reconstituted extract is examined by high performance liquid chromatography equipped with fluorescence detector.

| <b>PARAMETERS MEASURED :</b> | <b>LIS TEST CODE</b> | <b>W ( ng/L )</b> | <b>T ( ng/L )</b> |
|------------------------------|----------------------|-------------------|-------------------|
| phenanthrene                 | B3001X               | 10                | 100               |
| anthracene                   | B3002X               | 1                 | 10                |
| fluoranthene                 | B3003X               | 20                | 200               |
| pyrene                       | B3004X               | 20                | 200               |
| benzo(a)anthracene           | B3005X               | 20                | 200               |
| chrysene                     | B3006X               | 50                | 500               |
| dimethylbenz(a)anthracene    | B3007X               | 5                 | 50                |
| benzo(e)pyrene               | B3008X               | 50                | 500               |
| benzo(b)fluoranthene         | B3010X               | 10                | 100               |
| perylene                     | B3011X               | 10                | 100               |
| benzo(k)fluoranthene         | B3012X               | 1                 | 10                |
| benzo(a)pyrene               | B3013X               | 5                 | 50                |
| benzo(g,h,i)perylene         | B3014X               | 20                | 200               |
| dibenzo(a,h)anthracene       | B3015X               | 10                | 100               |
| indeno(1,2,3-c,d)pyrene      | B3016X               | 20                | 200               |
| benzo(b)chrysene             | B3017X               | 2                 | 20                |
| coronene                     | B3019X               | 10                | 100               |

**REPORTING FORMAT :**

Results are reported in parts per trillion (ng/L) rounded off to the closest increment of W and up to maximum of two significant figures.

**QUALITY CONTROL :**

The routine quality control operations monitor validity of calibration ( calibration check solution ), absence of potential interferences ( method blanks ), overall method performance ( fortified method blanks ).

For selected target compounds, control charts summarizing the response factors used to calibrate instruments and the recoveries from fortified method blanks are maintained.

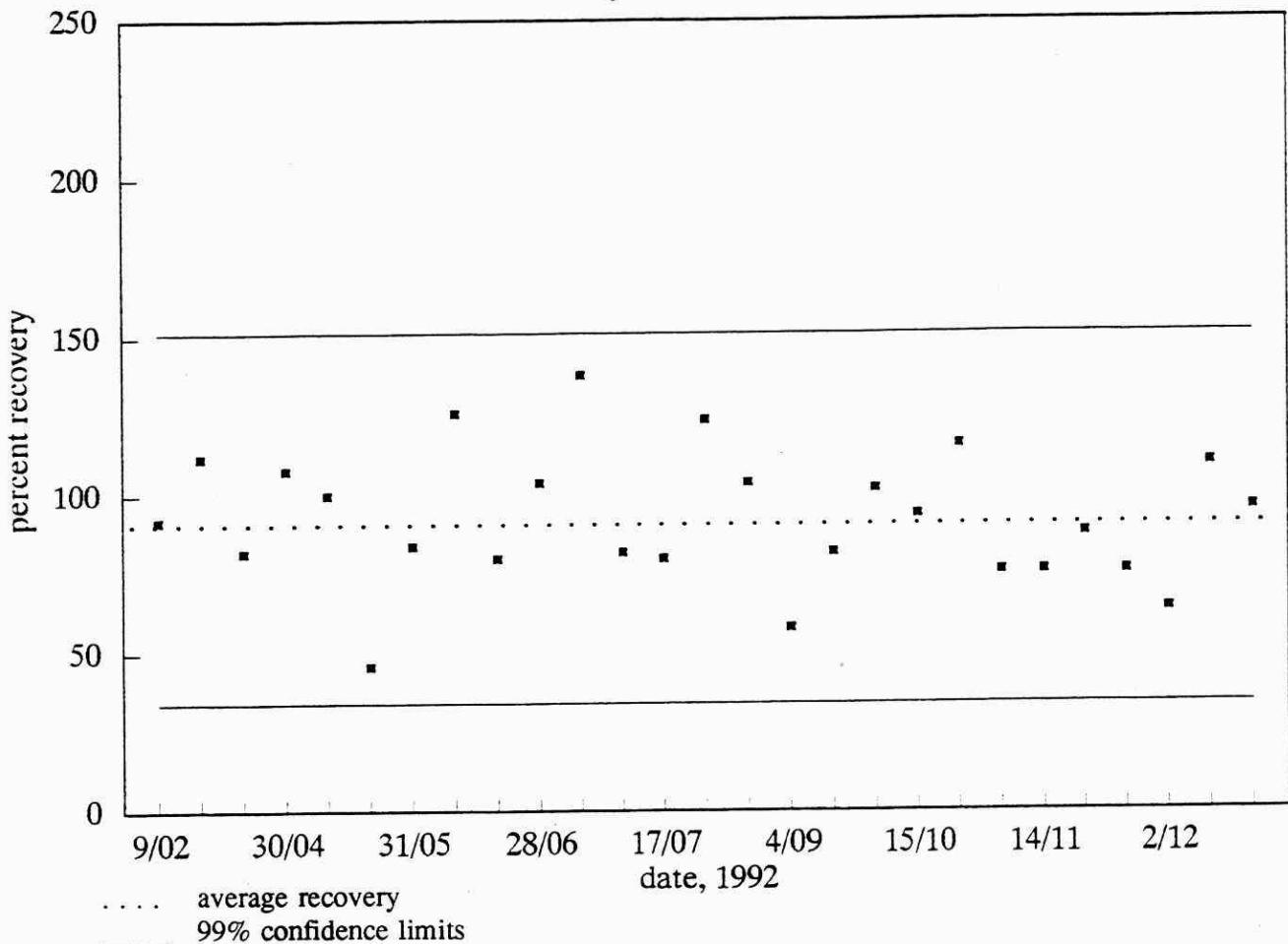
**REMARKS :** During the period starting January 1992 and ending December 1992, a total of 26 method blanks was prepared and tested by the method. For these 26 analyses, no observable responses of any of the target analytes were encountered.

List of Performance Charts :      Phenanthrene ( recovery from fortified blank )  
    Benzo(b)fluoranthene / Perylene ( recovery from fortified blank )  
    Benzo(a)pyrene ( recovery from fortified blank )

List of Performance Tables :      Phenanthrene  
    Benzo(b)fluoranthene / Perylene  
    Benzo(a)pyrene

# phenanthrene

recovery from fortified blank

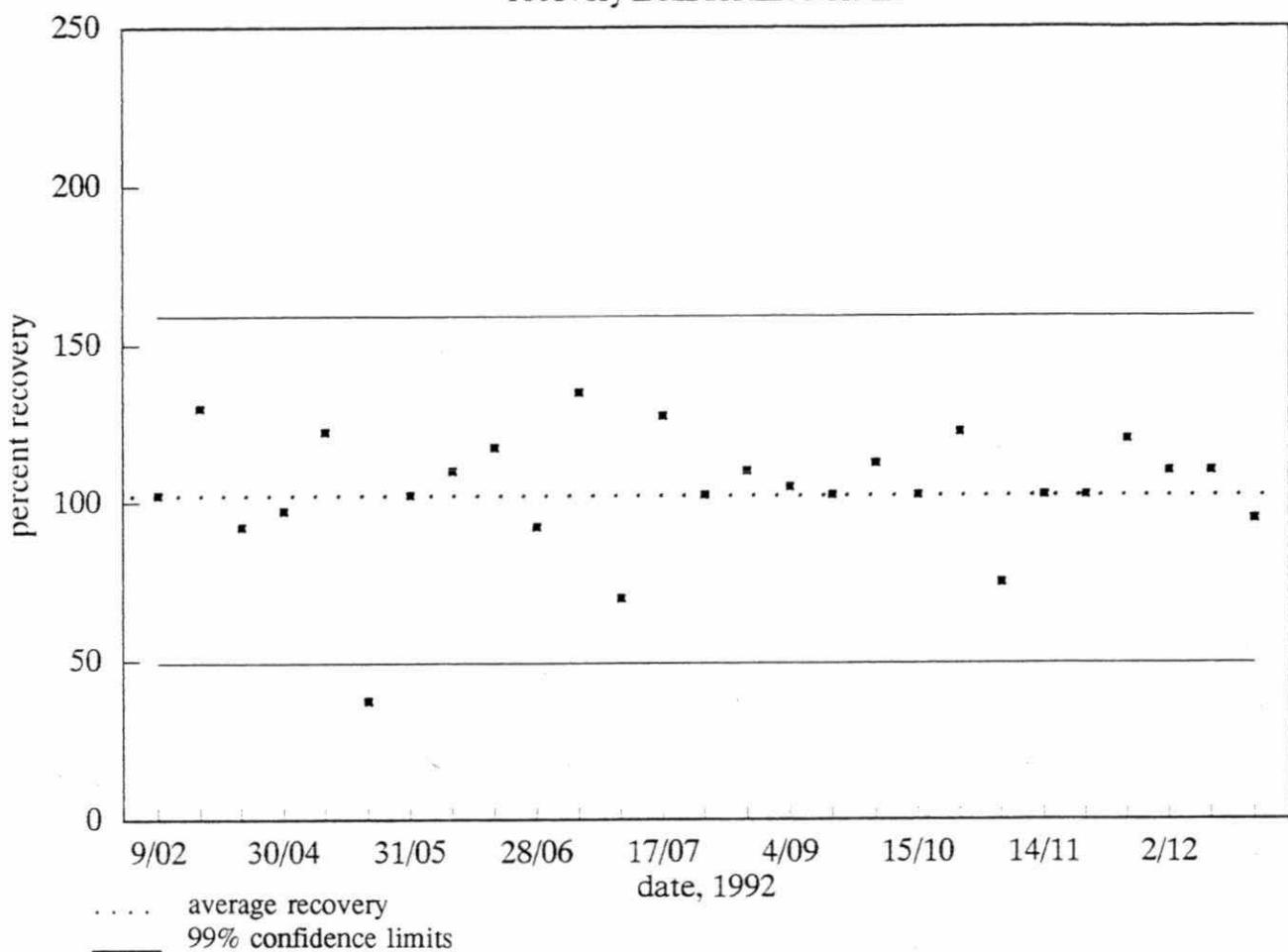


## Method Performance Summary

January - December 1992

|                                |              |
|--------------------------------|--------------|
| Analyte                        | phenanthrene |
| True Concentration             | 50 ng/L      |
| Number of Observations         | 27           |
| Between-run Standard Deviation | 21%          |
| Accuracy (% of expected)       | 93%          |

## benzo(b)fluoranthene/perylene



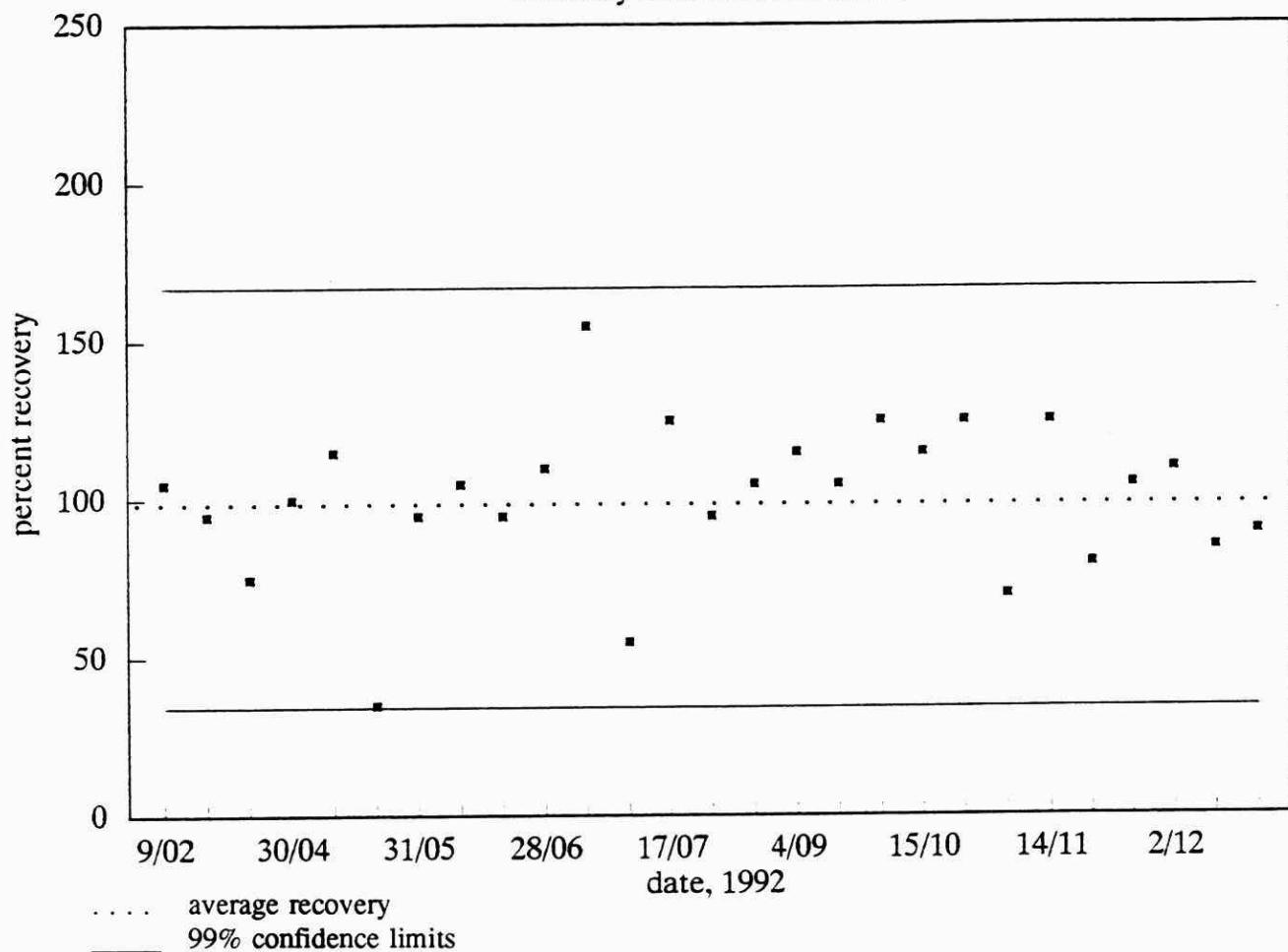
## Method Performance Summary

January - December 1992

|                                |                               |
|--------------------------------|-------------------------------|
| Analyte                        | benzo(b)fluoranthene/perylene |
| True Concentration             | 40 ng/L                       |
| Number of Observations         | 27                            |
| Between-run Standard Deviation | 20%                           |
| Accuracy (% of expected)       | 104%                          |

## benzo(a)pyrene

recovery from fortified blank



### Method Performance Summary

January - December 1992

|                                |                |
|--------------------------------|----------------|
| Analyte                        | benzo(a)pyrene |
| True Concentration             | 20 ng/L        |
| Number of Observations         | 27             |
| Between-run Standard Deviation | 24%            |
| Accuracy (% of expected)       | 101%           |

**METHOD CODE :** PWACAR-E3158A

**METHOD TITLE:** The Determination of Carbamates in Water by HPLC - UV Detection

**LABORATORY :** Organic Water Unit

**SUPERVISOR :** P. Crozier / Dr. D. Hall

**SAMPLE TYPE :** surface water, groundwater, raw and treated drinking water

**PRINCIPLE OF THE METHOD :**

Sample is extracted with an organic solvent; the extract is dried and evaporated to dryness. The reconstituted extract is examined by high performance liquid chromatography, using a variable wavelength ultraviolet detector.

| <b>PARAMETERS MEASURED :</b>       | <b>LIS TEST CODE</b> | <b>W ( ng/L )</b> | <b>T ( ng/L )</b> |
|------------------------------------|----------------------|-------------------|-------------------|
| carbofuran                         | P6CARB               | 2 000             | 20 000            |
| carbaryl                           | P6SEVN               | 200               | 2 000             |
| butylate                           | P6SUTN               | 2 000             | 20 000            |
| propoxur                           | P6PROP               | 2 000             | 20 000            |
| isopropyl-3-chlorophenyl carbamate | P6CIPC               | 2 000             | 20 000            |
| isopropyl phenyl carbamate         | P6IPC                | 2 000             | 20 000            |
| diallate                           | P6DIAL               | 2 000             | 20 000            |
| eptam                              | P6EPTM               | 2 000             | 20 000            |

**REPORTING FORMAT :**

Results are reported in parts per trillion (ng/L) rounded off to the closest increment of W up to maximum of two significant figures.

**QUALITY CONTROL :**

The routine quality control operations monitor validity of calibration ( calibration check solution ), absence of potential interferences ( method blanks ), overall method performance ( fortified method blanks ).

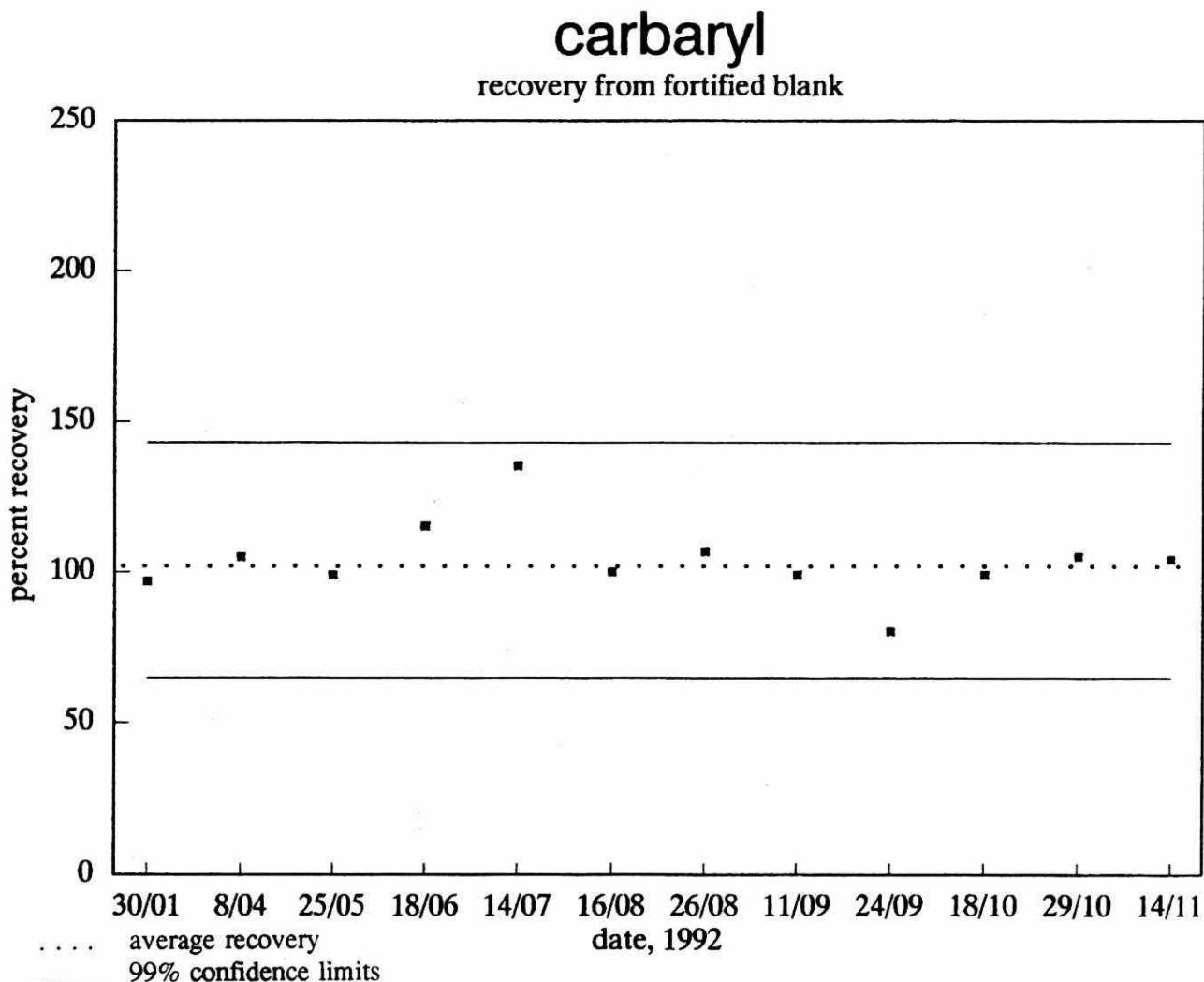
Control charts summarizing the response factors used to calibrate instruments and the recoveries from fortified method blanks are maintained for selected target compounds.

**REMARKS :** During the period starting January 1992 and ending December 1992, a total of 13 method blanks was prepared and tested by the method. For these 13 analyses, no observable responses of any of the target analytes were encountered.

In addition to the intra-laboratory method control, the performance of the method was examined through performance audit samples program organized by LSB Quality Management Office.

List of Performance Charts : Carbaryl ( recovery from fortified blank )  
Isopropyl-3-chlorophenyl Carbamate ( recovery from fortified blank )  
Butylate ( recovery from fortified blank )

List of Performance Tables : Carbaryl  
Isopropyl-3-chlorophenyl Carbamate  
Butylate



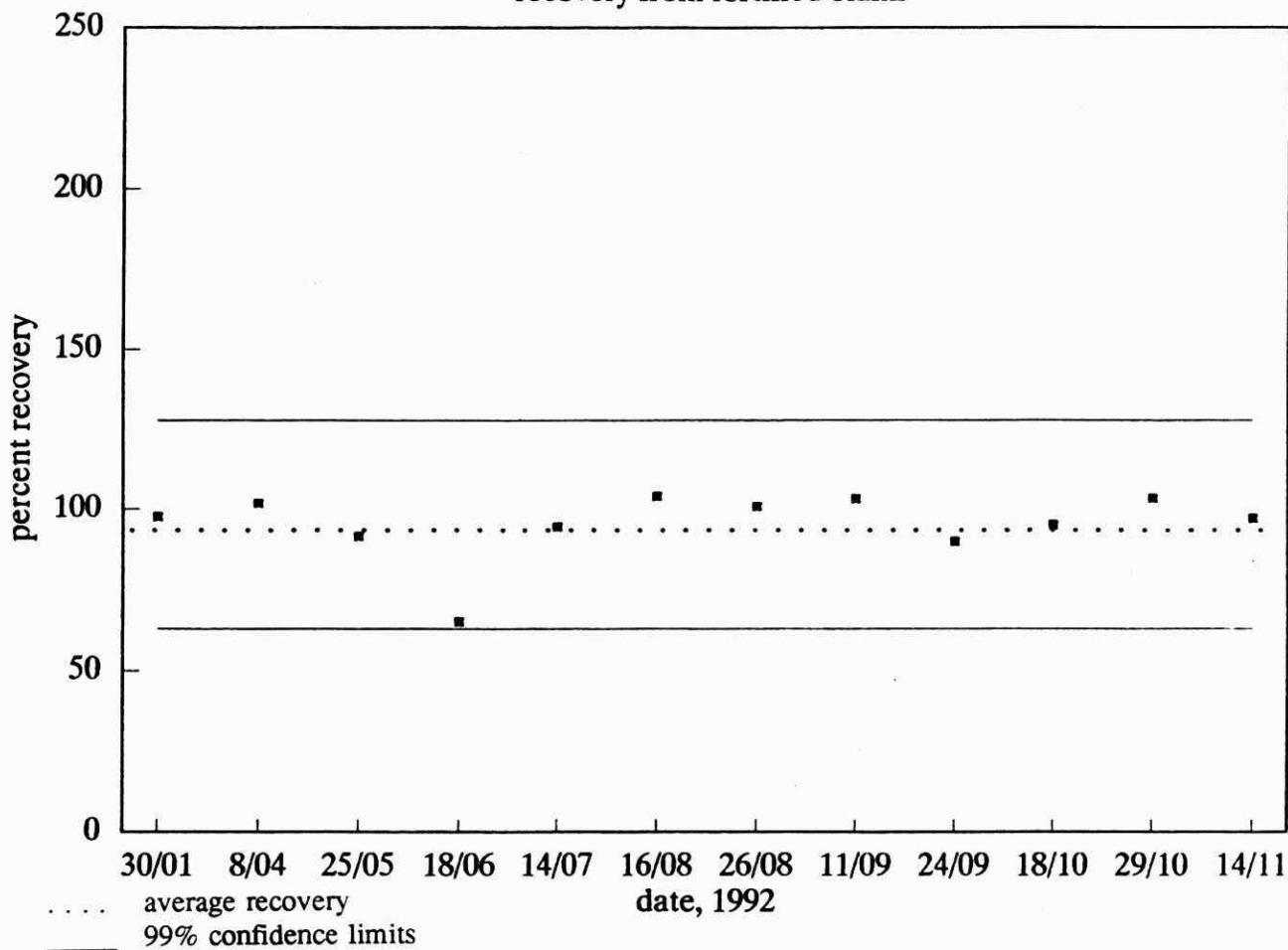
Method Performance Summary

January - December 1992

|                                |             |
|--------------------------------|-------------|
| Analyte                        | carbaryl    |
| True Concentration             | 10 000 ng/L |
| Number of Observations         | 12          |
| Between-run Standard Deviation | 13%         |
| Accuracy (% of expected)       | 104%        |

## isopropyl-3-chlorophenyl carbamate

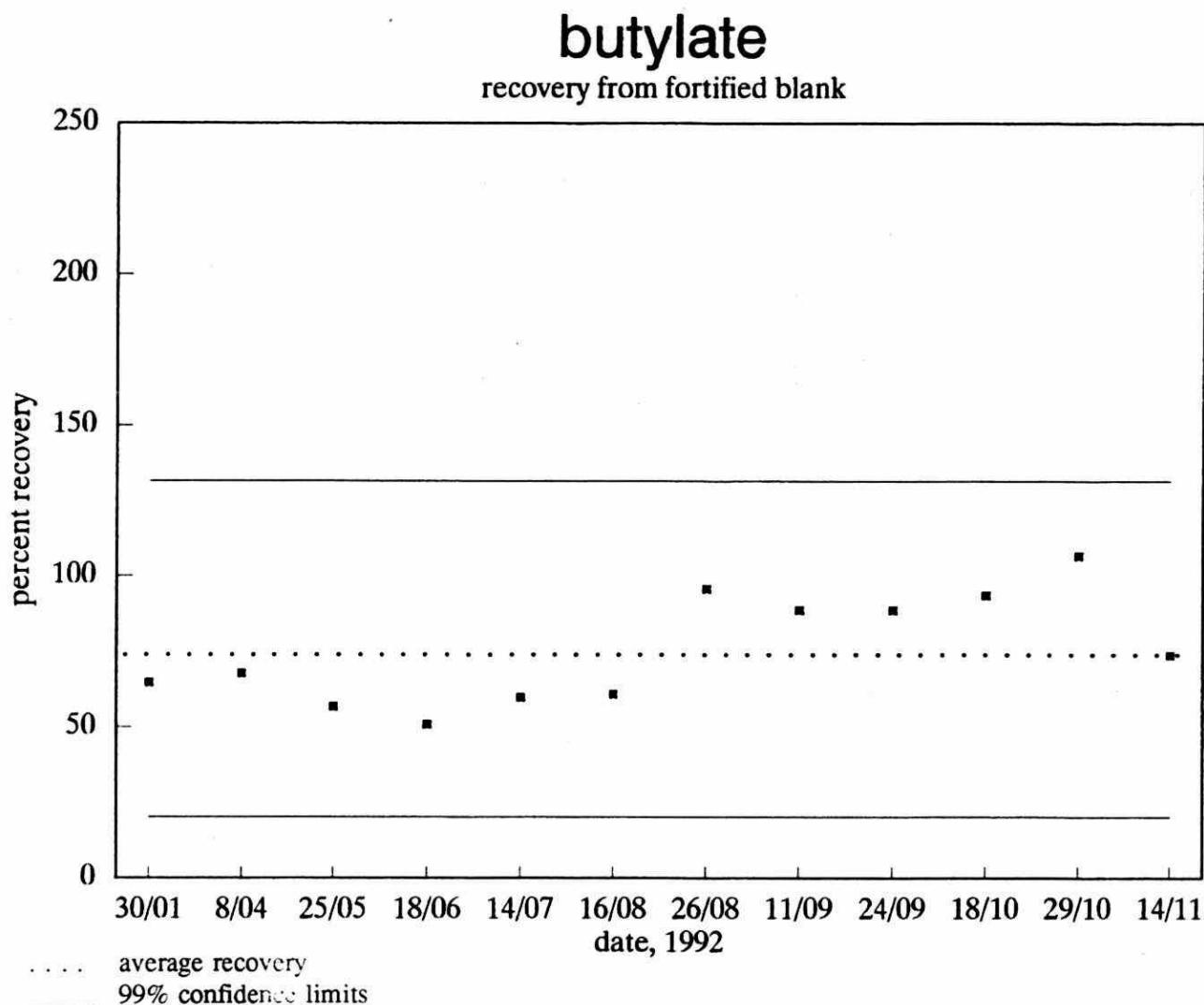
recovery from fortified blank



### Method Performance Summary

January - December 1992

|                                |                                    |
|--------------------------------|------------------------------------|
| Analyte                        | isopropyl-3-chlorophenyl carbamate |
| True Concentration             | 5 000 ng/L                         |
| Number of Observations         | 12                                 |
| Between-run Standard Deviation | 11%                                |
| Accuracy (% of expected)       | 95%                                |



**Method Performance Summary**

January - December 1992

|                                |            |
|--------------------------------|------------|
| Analyte                        | butylate   |
| True Concentration             | 5 000 ng/L |
| Number of Observations         | 12         |
| Between-run Standard Deviation | 18%        |
| Accuracy (% of expected)       | 76%        |

**METHOD CODE :** PWAUH-E3230A

**METHOD TITLE:** The Determination of Phenyl Ureas in Water by HPLC - UV Detection

**LABORATORY :** Organic Water Unit

**SUPERVISOR :** P. Crozier / Dr. D. Hall

**SAMPLE TYPE :** surface water, groundwater, raw and treated drinking water

**PRINCIPLE OF THE METHOD :**

Samples are extracted with an organic solvent; the extract is filtered through granular anhydrous sodium sulphate to remove water and evaporated to dryness by rotary evaporator. The reconstituted extract is examined by high performance liquid chromatography using a variable wavelength ultraviolet detector.

| <b>PARAMETERS MEASURED :</b> | <b>LIS TEST CODE</b> | <b>W ( ng/L )</b> | <b>T ( ng/L )</b> |
|------------------------------|----------------------|-------------------|-------------------|
| linuron                      | P5LINU               | 2 000             | 20 000            |
| monuron                      | P5MONU               | 2 000             | 20 000            |
| diuron                       | P5DIUR               | 2 000             | 20 000            |
| chlortoluron                 | P5CTOL               | 2 000             | 20 000            |
| fluometuron                  | P5FMET               | 2 000             | 20 000            |
| monolinuron                  | P5MLIN               | 2 000             | 20 000            |
| chlorbromuron                | P5CBRO               | 2 000             | 20 000            |
| metoxuron                    | P5METX               | 2 000             | 20 000            |
| siduron                      | P5SID                | 2 000             | 20 000            |
| difenoxyuron                 | P5DIF                | 2 000             | 20 000            |
| neburon                      | P5NEB                | 2 000             | 20 000            |
| metobromuron                 | P5PATO               | 2 000             | 20 000            |

**REPORTING FORMAT :**

Results are reported in parts per trillion (ng/L) rounded off to the closest increment of 100 ng/L and up to maximum of two significant figures.

**QUALITY CONTROL :**

The routine quality control operations monitor validity of calibration ( calibration check solution ), absence of potential interferences ( method blanks ), overall method performance ( fortified method blanks ).

**REMARKS :** During the period starting January 1991 and ending December 1991, one method blank was prepared and tested by the method. No observable responses of any of the target analytes were encountered.

Since this method is not used on regular basis ( less than 50 samples are analyzed per year ), no control charts are maintained.

List of Performance Charts : not applicable

List of Performance Tables : Recoveries of Target Analytes from Fortified Method Blanks

Performance Summary Table

Recoveries of PWAUH Target Analytes from Fortified Method Blanks

| Analyte              | concentration<br>( ng/L ) | number<br>of obs. | accuracy<br>( % of expected ) |
|----------------------|---------------------------|-------------------|-------------------------------|
| metoxuron            | 5 000                     | 1                 | 93%                           |
| monuron              | 5 000                     | 1                 | 90%                           |
| chlortoluron         | 5 000                     | 1                 | 101%                          |
| fluometuron          | 5 000                     | 1                 | 89%                           |
| diuron / monolinuron | 10 000                    | 1                 | 101%                          |
| difenoxyuron         | 5 000                     | 1                 | 98%                           |
| metobromuron         | 5 000                     | 1                 | 96%                           |
| siduron              | 5 000                     | 1                 | 96%                           |
| linuron              | 5 000                     | 1                 | 102%                          |
| chlorbromuron        | 5 000                     | 1                 | 101%                          |
| neburon              | 5 000                     | 1                 | 100%                          |

**METHOD CODE :** NDMA-E3291A

**METHOD TITLE:** The Determination of N-Nitrosodimethylamine (NDMA) in Drinking Water and in Aqueous Samples by Gas Chromatography / High Resolution Mass Spectrometry (GC/HRMS)

**LABORATORY :** Mass Spectrometry Unit

**SUPERVISOR :** Dr. V. Taguchi

**SAMPLE TYPE :** drinking water, aqueous samples

**PRINCIPLE OF THE METHOD :**

The internal standard d<sub>6</sub>-NDMA is added to an aliquot of the sample. After the pH is adjusted to 12 to keep the acidic components in the aqueous phase, the basic solution is serially extracted with dichloromethane. The dichloromethane extract is washed with a sulphuric acid solution to remove basic components from the organic phase. After being filtered through granular anhydrous sodium sulphate to remove water the extract is concentrated by rotary evaporator and a nitrogen evaporating unit.

The sample extract is analysed by GC/HRMS. NDMA is quantified by an isotope dilution method.

**PARAMETERS MEASURED :**

**LIS TEST CODE**

**MDL (µg/L)**

N-Nitrosodimethylamine

MSNDMA

0.005

**REPORTING FORMAT :**

Results are reported in µg/L rounded off to two significant figures. The lowest reported value is 5x10<sup>-3</sup>µg/L.

**QUALITY CONTROL :**

The routine quality control operations monitor overall method performance ( spiked procedure blanks ), size of potential positive bias ( method blanks ) and maintenance of the required instrument sensitivity.

**REMARKS :** The analytical method was modified in October 1992. Single point calibration was replaced with a multi-point calibration.

In addition to the intra-laboratory method control, the performance of the method was examined through the performance audit samples program organized by the LSB Quality Management Office.

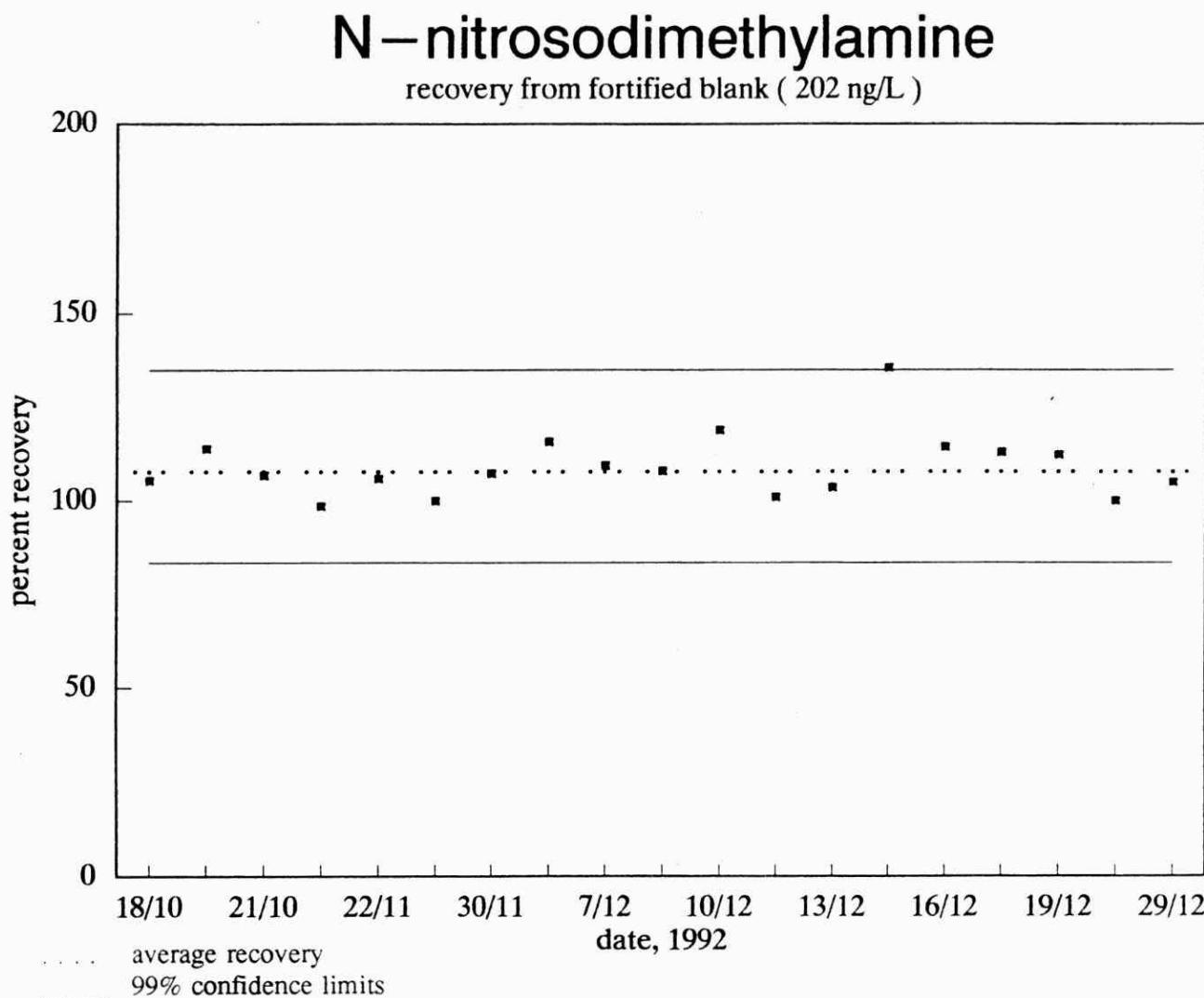
List of Performance Charts and Tables:

N-Nitrosodimethylamine ( method blanks summary)  
N-Nitrosodimethylamine ( recovery from spiked blanks - 0.0104 µg/L )  
N-Nitrosodimethylamine ( recovery from spiked blanks - 0.202 µg/L )

**Method Blanks Summary**

January - December 1992

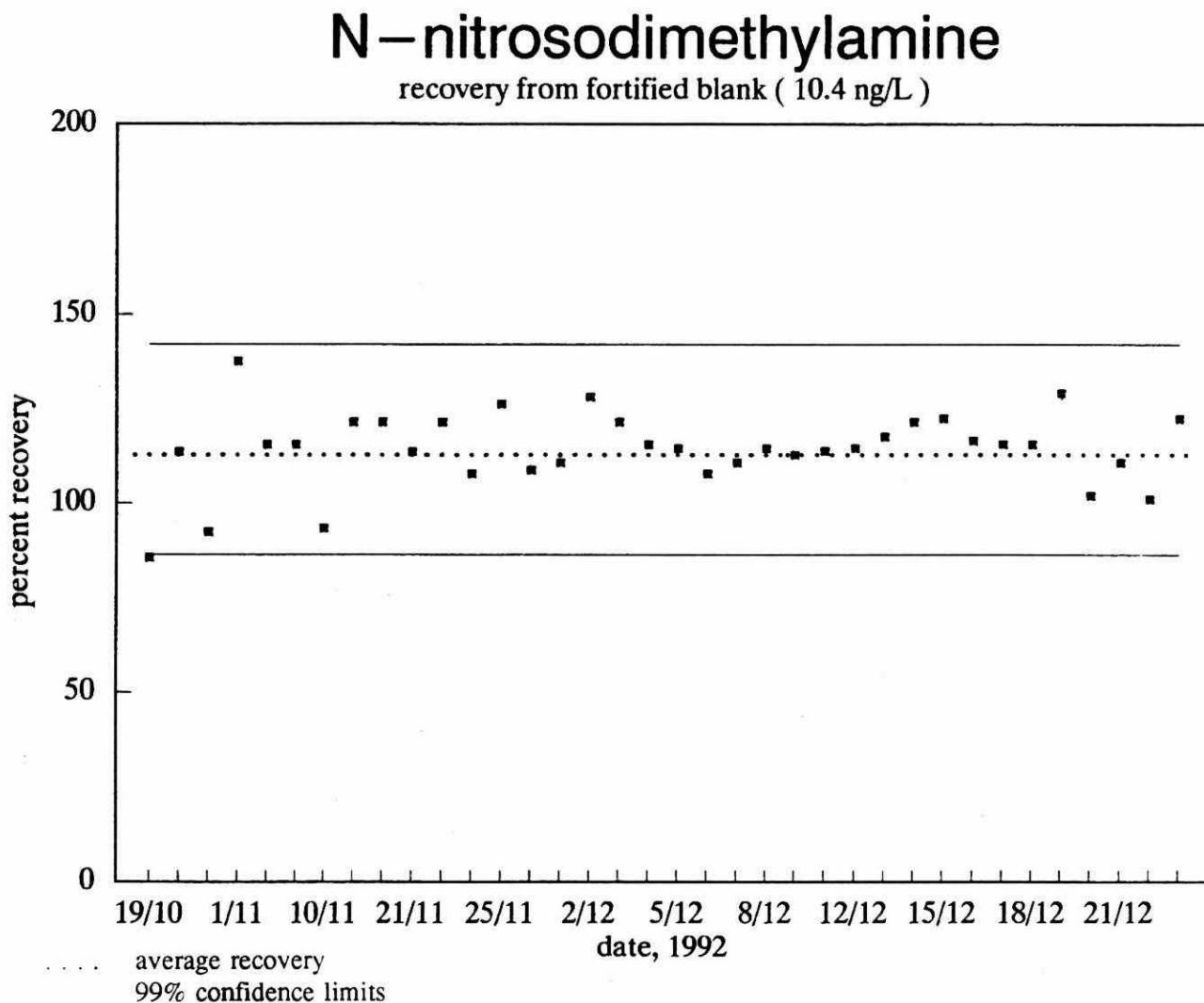
| Analyte                | N-nitrosodimethylamine    |
|------------------------|---------------------------|
| Number of Observations | 183                       |
| Mean Concentration     | $1.4 \times 10^{-3}$ µg/L |
| Standard Deviation     | $1.1 \times 10^{-3}$ µg/L |



#### Method Performance Summary

January - December 1992

|                                |                        |
|--------------------------------|------------------------|
| Analyte                        | N-nitrosodimethylamine |
| True Concentration             | 0.202 µg/L             |
| Number of Observations         | 19                     |
| Between-run Standard Deviation | 8%                     |
| Accuracy (% of expected)       | 109%                   |



#### Method Performance Summary

January - December 1992

|                                |                        |
|--------------------------------|------------------------|
| Analyte                        | N-nitrosodimethylamine |
| True Concentration             | 0.0104 µg/L            |
| Number of Observations         | 36                     |
| Between-run Standard Deviation | 10%                    |
| Accuracy (% of expected)       | 114%                   |

**METHOD CODE :** PAAFD-E3122A

**METHOD TITLE:** The Determination of Polychlorinated Dibeno-p-dioxins (PCDD) and Polychlorinated Dibenzofurans (PCDF) in Ambient Air

**LABORATORY :** Dioxin Unit

**SUPERVISOR :** Dr. E. Reiner

**SAMPLE TYPE :** ambient air

**PRINCIPLE OF THE METHOD :**

Samples are collected using a MOE-modified high-volume air sampler with a polyurethane foam (PUF) plug and Teflon-coated glass fibre filter. A known quantity of isotopically labelled PCDDs and PCDFs is added to each sample to serve as an internal quantitation standard. PCDDs and PCDFs are extracted from the PUF and filter using a Soxhlet extraction apparatus and toluene. The concentrated extract is processed through a multi-stage chromatographic cleanup procedure to remove potential chemical interferences. After cleanup, the extract is evaporated to dryness.

The reconstituted extract is analyzed by gas chromatography - tandem mass spectrometry (GC-MS-MS) or gas chromatography - high resolution mass spectrometry (GC-HRMS).

Further cleanup using high performance liquid chromatography (HPLC) may be necessary prior to final analysis if the sample is highly contaminated with chemical interferences that are not removed by the open-column chromatographic cleanup.

| <b>PARAMETERS MEASURED :</b>              | <b>IDL ( pg/m<sup>3</sup> )</b> | <b>MDL ( pg/m<sup>3</sup> )</b> |
|---|---------------------------------|---------------------------------|
| 2,3,7,8-tetrachlorodibenzo-p-dioxin       | 0.001                           | 0.005                           |
| 1,2,3,7,8-pentachlorodibenzo-p-dioxin     | 0.001                           | 0.02                            |
| 1,2,3,4,7,8-hexachlorodibenzo-p-dioxin    | 0.002                           | 0.02                            |
| 1,2,3,6,7,8-hexachlorodibenzo-p-dioxin    | 0.002                           | 0.02                            |
| 1,2,3,7,8,9-hexachlorodibenzo-p-dioxin    | 0.002                           | 0.02                            |
| 1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin | 0.003                           | 0.02                            |
| octachlorodibenzo-p-dioxin                | 0.005                           | 0.06                            |
| 2,3,7,8-tetrachlorodibenzofuran           | 0.001                           | 0.007                           |
| 2,3,4,7,8-pentachlorodibenzofuran         | 0.001                           | 0.02                            |
| 1,2,3,7,8-pentachlorodibenzofuran         | 0.001                           | 0.02                            |
| 1,2,3,4,7,8-hexachlorodibenzofuran        | 0.002                           | 0.02                            |
| 1,2,3,6,7,8-hexachlorodibenzofuran        | 0.002                           | 0.01                            |
| 2,3,4,6,7,8-hexachlorodibenzofuran        | 0.002                           | 0.02                            |
| 1,2,3,7,8,9-hexachlorodibenzofuran        | 0.002                           | 0.02                            |
| 1,2,3,4,6,7,8-heptachlorodibenzofuran     | 0.003                           | 0.04                            |
| 1,2,3,4,7,8,9-heptachlorodibenzofuran     | 0.003                           | 0.03                            |
| octachlorodibenzofuran                    | 0.005                           | 0.07                            |

( parameters measured continued )

total tetrachlorinated dibenzo-p-dioxins ( TCDD )  
total pentachlorinated dibenzo-p-dioxins ( PCDD )  
total hexachlorinated dibenzo-p-dioxins ( HxCDD )  
total heptachlorinated dibenzo-p-dioxins ( HpCDD )  
total tetrachlorinated dibenzofurans ( TCDF )  
total pentachlorinated dibenzofurans ( PCDF )  
total hexachlorinated dibenzofurans ( HxCDF )  
total heptachlorinated dibenzofurans ( HpCDF )

**REPORTING FORMAT :**

Results are reported as pg/m<sup>3</sup> rounded off to 2 significant figures. The minimum reported levels are sample and analyte specific and range from 0.001 pg/m<sup>3</sup> to 0.01 pg/m<sup>3</sup>.

**QUALITY CONTROL :**

The routine quality control operations monitor overall method performance ( precision and recovery samples ), validity of calibration and consistency in injection volume ( injection standard ), absence of potential contamination ( method blanks ) and recovery of target analytes ( internal quantitation standard ).

**REMARKS :** Two types of performance limits are displayed on the performance charts. One set was statistically derived from the 1992 data; while the other ( established at recoveries of 70% and 130% ) was adopted by the Dioxin Unit as method performance control limits.

List of Performance Charts and Tables:

Method Blanks Summary  
2,3,7,8-tetrachlorodibenzo-p-dioxin  
1,2,3,7,8-pentachlorodibenzo-p-dioxin  
1,2,3,4,7,8-hexachlorodibenzo-p-dioxin  
1,2,3,6,7,8-hexachlorodibenzo-p-dioxin  
1,2,3,7,8,9-hexachlorodibenzo-p-dioxin  
1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin  
octachlorodibenzo-p-dioxin  
2,3,7,8-tetrachlorodibenzofuran  
2,3,4,7,8-pentachlorodibenzofuran  
1,2,3,7,8-pentachlorodibenzofuran  
1,2,3,4,7,8-hexachlorodibenzofuran

---

The minimum reported levels correspond to the amount of analyte that would give most-abundant ion response five times higher than corresponding instrumental noise.

( List of Performance Charts and Tables )

1,2,3,6,7,8-hexachlorodibenzofuran  
2,3,4,6,7,8-hexachlorodibenzofuran  
1,2,3,7,8,9-hexachlorodibenzofuran  
1,2,3,4,6,7,8-heptachlorodibenzofuran  
1,2,3,4,7,8,9-heptachlorodibenzofuran  
octachlorodibenzofuran

Method Blanks Summary

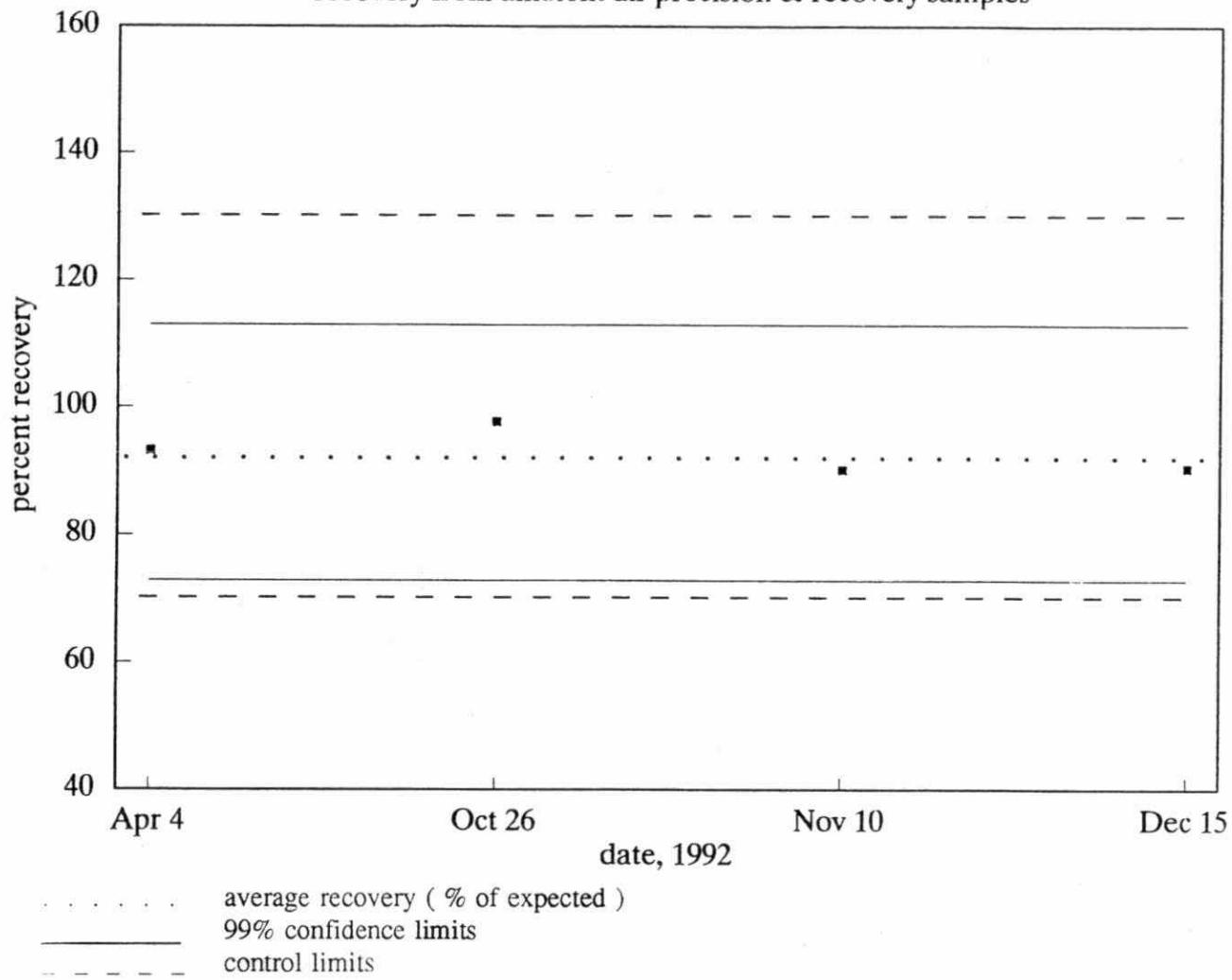
January 1992 - December 1992

| Analyte                                   | Number of Observations | Average Concentration ( $\times 10^3$ pg/m <sup>3</sup> ) | Standard Deviation ( $\times 10^3$ pg/m <sup>3</sup> ) |
|---|------------------------|---|--|
| 2,3,7,8-tetrachlorodibenzo-p-dioxin       | 13                     | ND ( 1 )  |  |
| 1,2,3,7,8-pentachlorodibenzo-p-dioxin     | 13                     | 0.23  | 0.80   |
| 1,2,3,4,7,8-hexachlorodibenzo-p-dioxin    | 13                     | ND ( 2 )  |  |
| 1,2,3,6,7,8-hexachlorodibenzo-p-dioxin    | 13                     | 0.3   | 1.1  |
| 1,2,3,7,8,9-hexachlorodibenzo-p-dioxin    | 13                     | 0.4   | 1.6  |
| 1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin | 13                     | 1.5   | 5.3  |
| octachlorodibenzo-p-dioxin                | 13                     | 6   | 14   |
| 2,3,7,8-tetrachlorodibenzofuran           | 13                     | 2.9   | 9.8  |
| 2,3,4,7,8-pentachlorodibenzofuran         | 13                     | 0.5   | 1.6  |
| 1,2,3,7,8-pentachlorodibenzofuran         | 13                     | 0.8   | 2.7  |
| 1,2,3,4,7,8-hexachlorodibenzofuran        | 13                     | 1.2   | 4.7  |
| 1,2,3,6,7,8-hexachlorodibenzofuran        | 13                     | 0.5   | 1.6  |
| 2,3,4,6,7,8-hexachlorodibenzofuran        | 13                     | 0.8   | 2.4  |
| 1,2,3,7,8,9-hexachlorodibenzofuran        | 13                     | ND ( 2 )  |  |
| 1,2,3,4,6,7,8-heptachlorodibenzofuran     | 13                     | 1.9   | 4.6  |
| 1,2,3,4,7,8,9-heptachlorodibenzofuran     | 13                     | ND ( 3 )  |  |
| octachlorodibenzofuran                    | 13                     | 0.9   | 2.9  |

ND ... Not detected. Detection limit in fg/m<sup>3</sup> given in brackets ( ).

## 2,3,7,8-tetrachlorodibenzo-p-dioxin

recovery from ambient air precision & recovery samples



Performance Summary Table

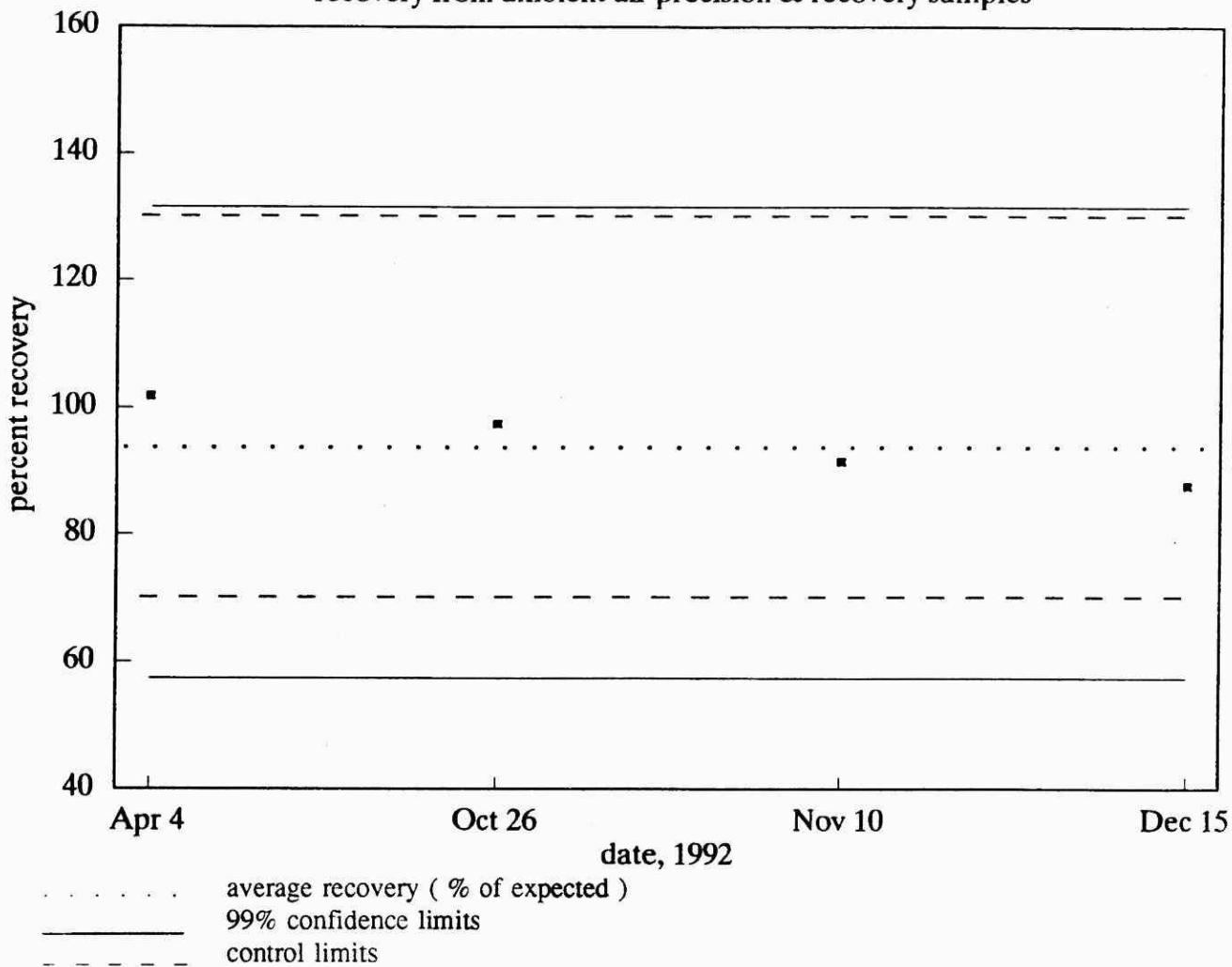
January - December 1992

|                                |                                     |
|--------------------------------|-------------------------------------|
| Analyte                        | 2,3,7,8-tetrachlorodibenzo-p-dioxin |
| True Concentration             | 0.27 pg/m <sup>3</sup> *            |
| Number of Observations         | 4                                   |
| Between-run Standard Deviation | 3.4 %                               |
| Accuracy (% of expected)       | 92.9 %                              |

true concentration relates to the original sample volume of 3 000 m<sup>3</sup>; see official text of the method for the details on spiking procedure

## 1,2,3,7,8-pentachlorodibenzo-p-dioxin

recovery from ambient air precision & recovery samples



Performance Summary Table

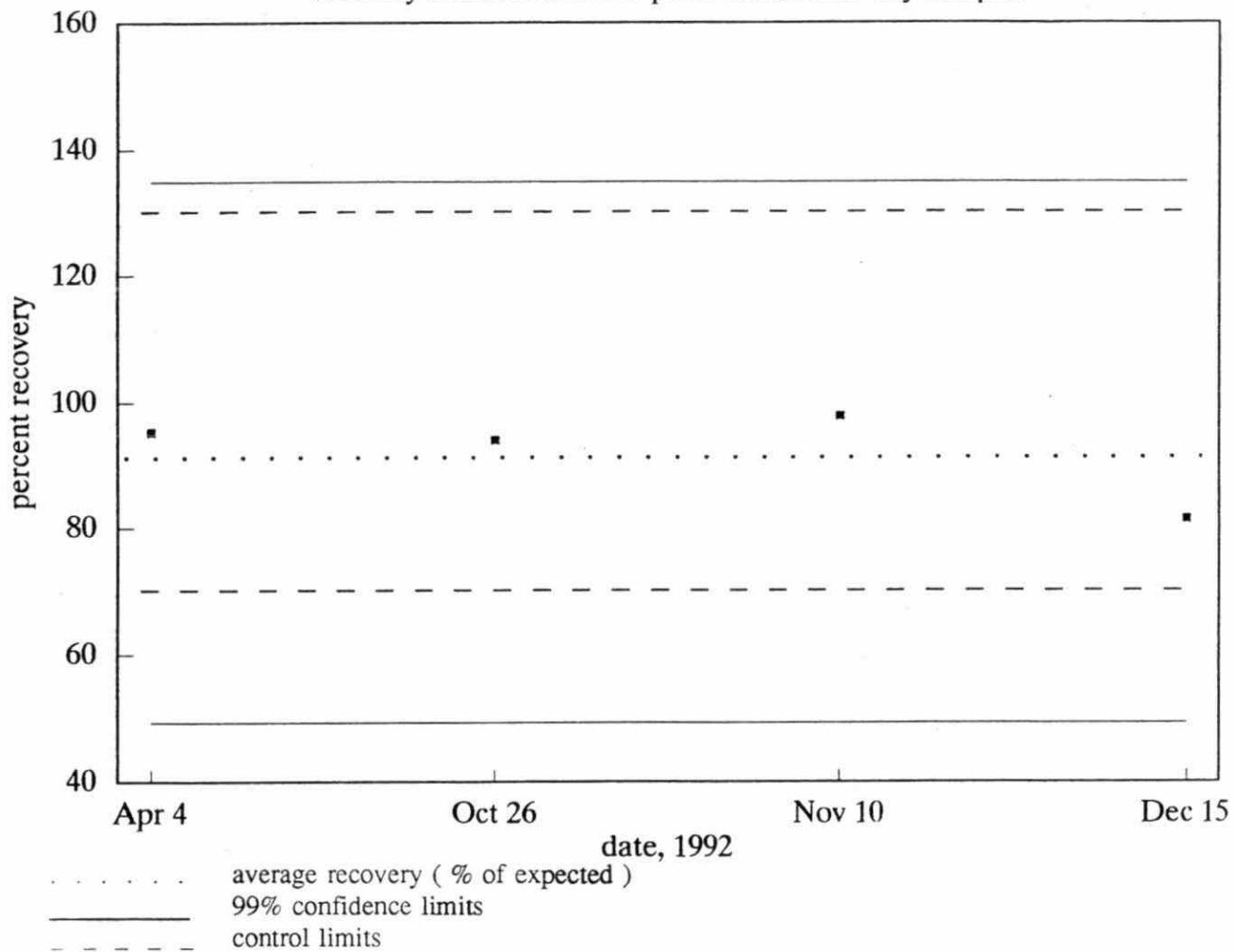
January - December 1992

|                                |                                       |
|--------------------------------|---------------------------------------|
| Analyte                        | 1,2,3,7,8-pentachlorodibenzo-p-dioxin |
| True Concentration             | 1.3 pg/m <sup>3</sup> *               |
| Number of Observations         | 4                                     |
| Between-run Standard Deviation | 6.4 %                                 |
| Accuracy (% of expected)       | 94.5 %                                |

\* true concentration relates to the original sample volume of 3 000 m<sup>3</sup>; see official text of the method for the details on spiking procedure

## 1,2,3,4,7,8-hexachlorodibenzo-p-dioxin

recovery from ambient air precision & recovery samples



Performance Summary Table

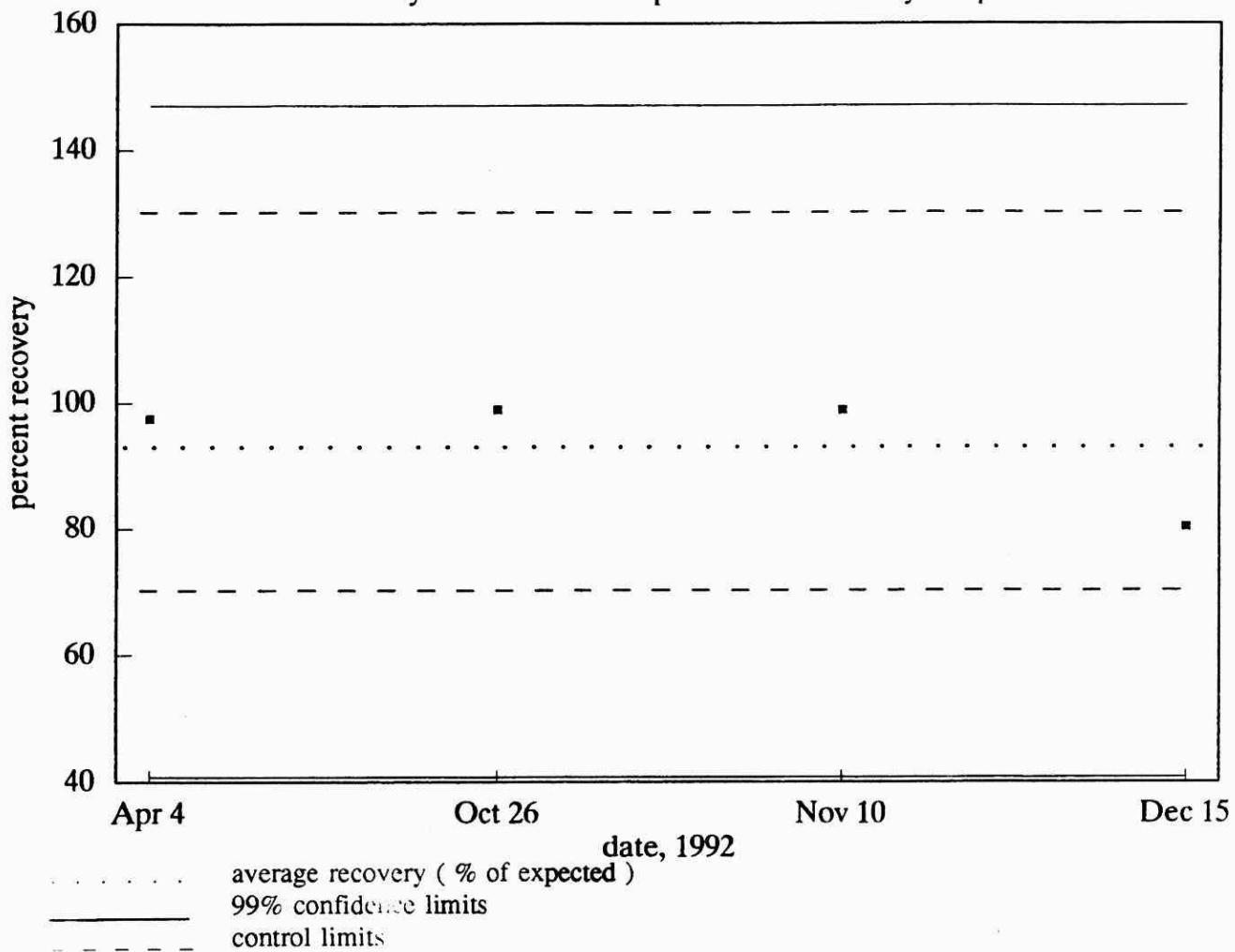
January - December 1992

|                                |  |
|--------------------------------|--|
| Analyte                        | 1,2,3,4,7,8-hexachlorodibenzo-p-dioxin |
| True Concentration             | 1.3 pg/m <sup>3</sup> *                |
| Number of Observations         | 4                                      |
| Between-run Standard Deviation | 7 %                                    |
| Accuracy (% of expected)       | 92 %                                   |

true concentration relates to the original sample volume of 3 000 m<sup>3</sup>; see official text of the method for the details on spiking procedure

## 1,2,3,6,7,8-hexachlorodibenzo-p-dioxin

recovery from ambient air precision & recovery samples



Performance Summary Table

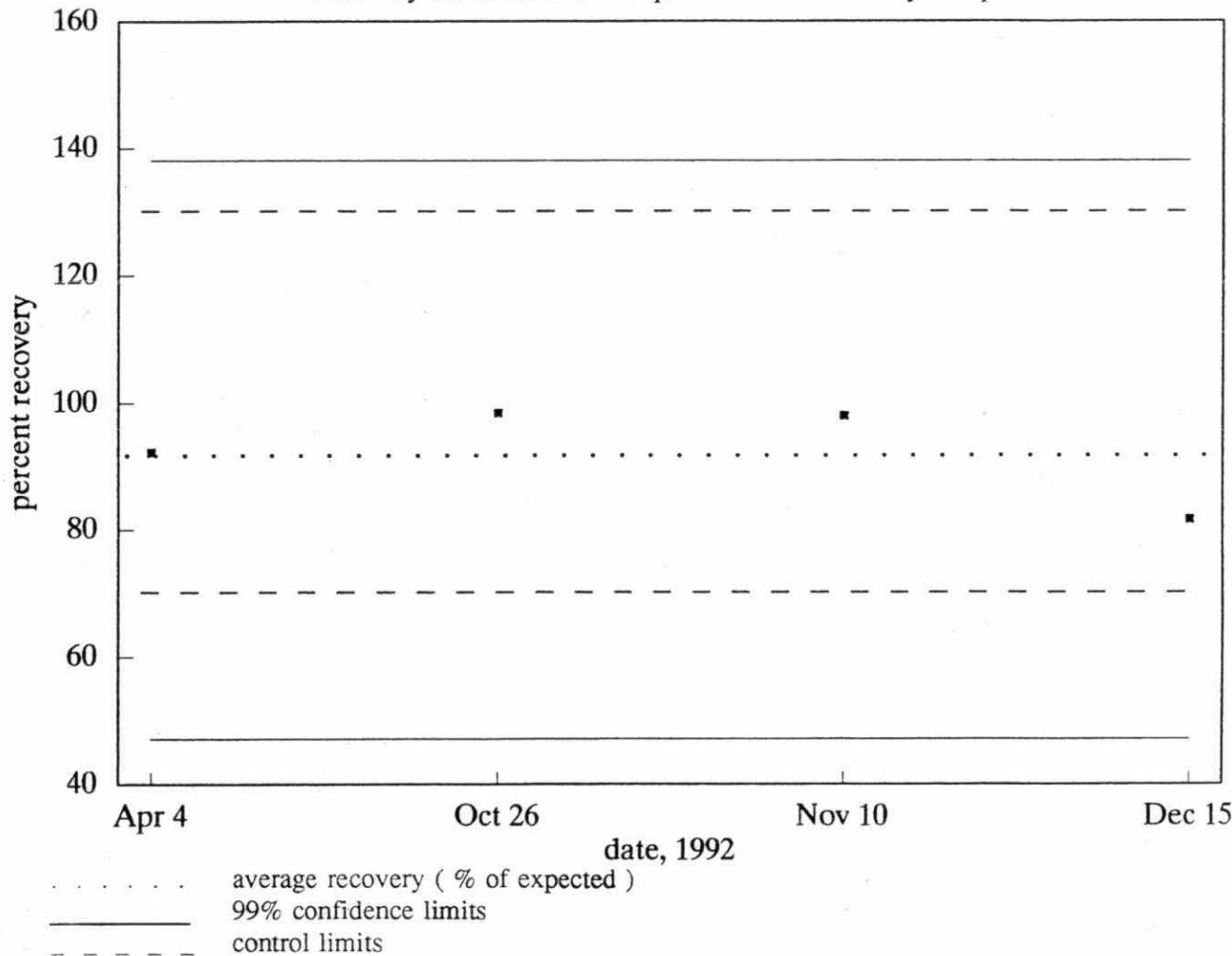
January - December 1992

|                                |  |
|--------------------------------|--|
| Analyte                        | 1,2,3,6,7,8-hexachlorodibenzo-p-dioxin |
| True Concentration             | 1.3 pg/m <sup>3</sup> *                |
| Number of Observations         | 4                                      |
| Between-run Standard Deviation | 9 %                                    |
| Accuracy (% of expected)       | 94 %                                   |

\* true concentration relates to the original sample volume of 3 000 m<sup>3</sup>; see official text of the method for the details on spiking procedure

## 1,2,3,7,8,9-hexachlorodibenzo-p-dioxin

recovery from ambient air precision & recovery samples



Performance Summary Table

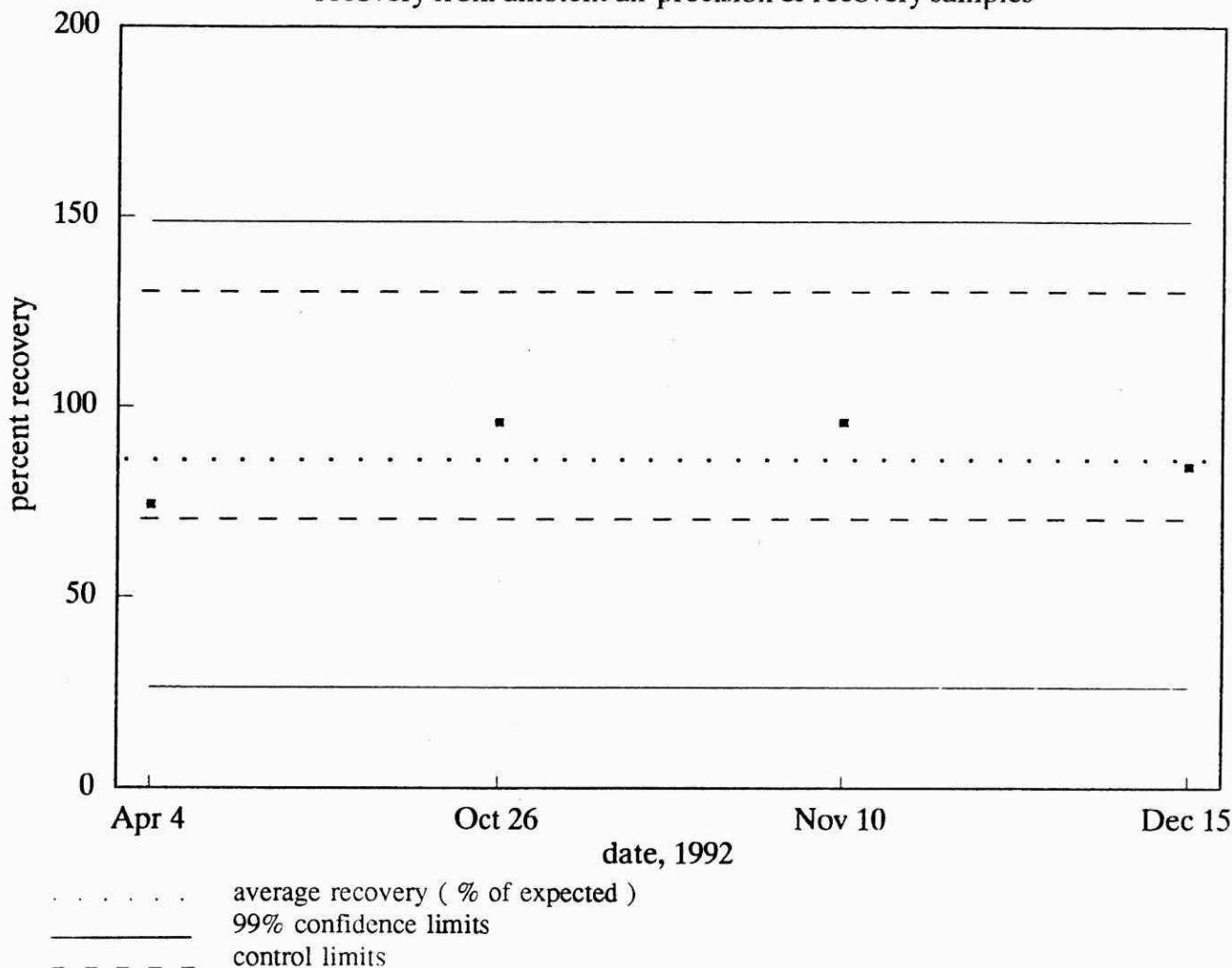
January - December 1992

|                                |  |
|--------------------------------|--|
| Analyte                        | 1,2,3,7,8,9-hexachlorodibenzo-p-dioxin |
| True Concentration             | 1.3 pg/m <sup>3</sup> *                |
| Number of Observations         | 4                                      |
| Between-run Standard Deviation | 8 %                                    |
| Accuracy (% of expected)       | 93 %                                   |

\* true concentration relates to the original sample volume of 3 000 m<sup>3</sup>; see official text of the method for the details on spiking procedure

# 1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin

recovery from ambient air precision & recovery samples



Performance Summary Table

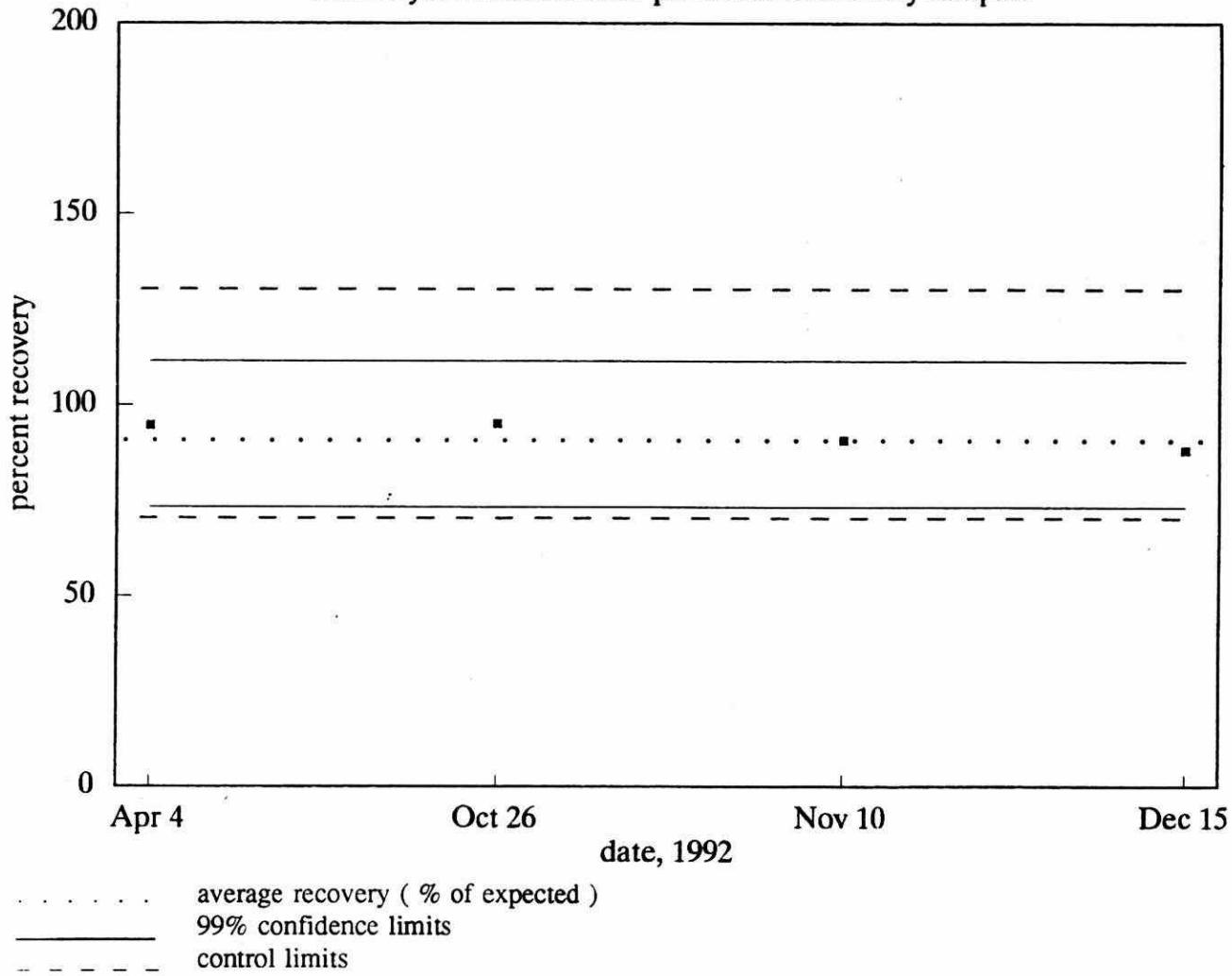
January - December 1992

|                                |   |
|--------------------------------|---|
| Analyte                        | 1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin |
| True Concentration             | 1.3 pg/m <sup>3</sup> *                   |
| Number of Observations         | 4   |
| Between-run Standard Deviation | 11 %                                      |
| Accuracy (% of expected)       | 88 %                                      |

\* true concentration relates to the original sample volume of 3 000 m<sup>3</sup>; see official text of the method for the details on spiking procedure

## octachlorodibenzo-p-dioxin

recovery from ambient air precision & recovery samples



Performance Summary Table

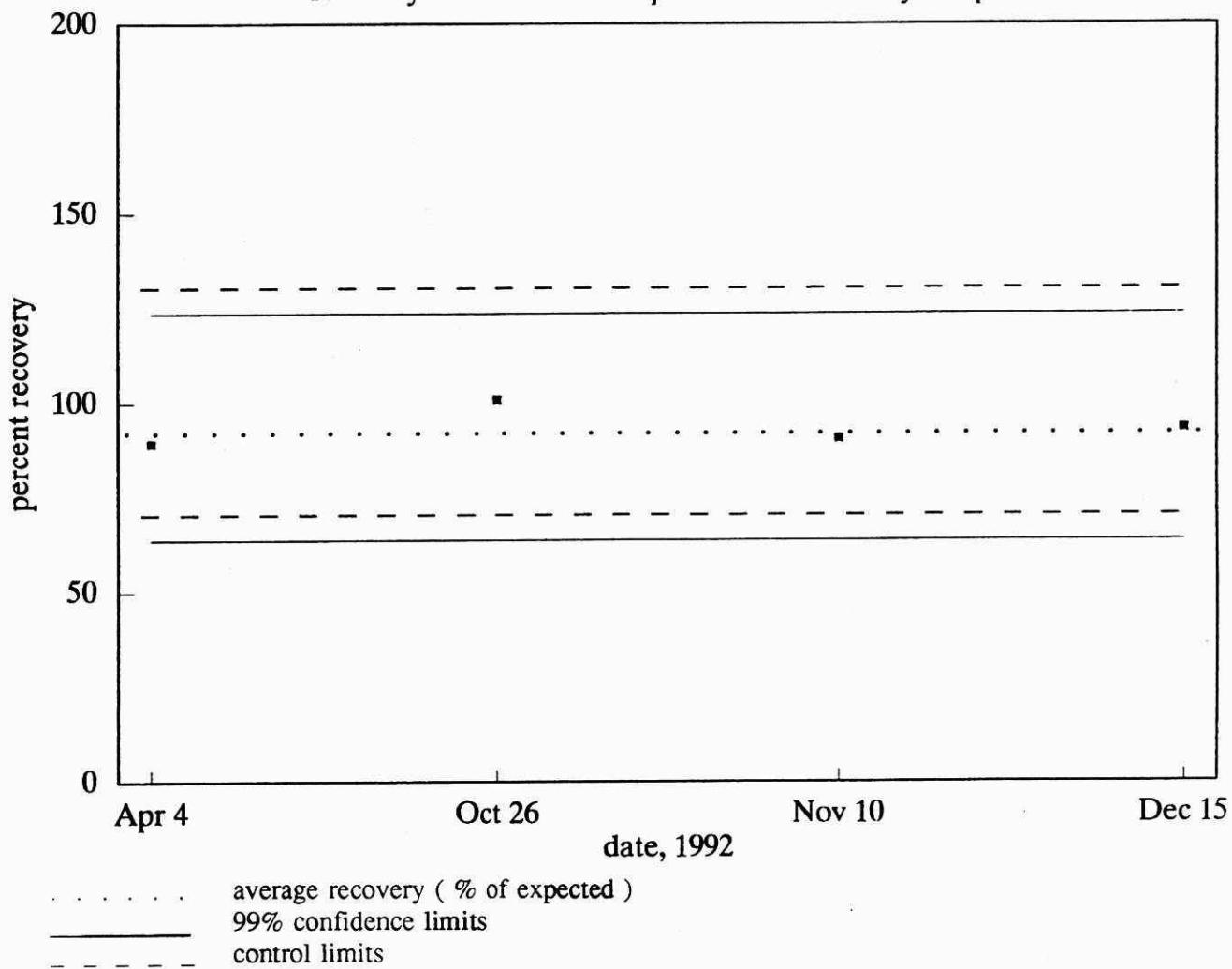
January - December 1992

|                                |                            |
|--------------------------------|----------------------------|
| Analyte                        | octachlorodibenzo-p-dioxin |
| True Concentration             | 2.7 pg/m <sup>3</sup> *    |
| Number of Observations         | 4                          |
| Between-run Standard Deviation | 3.3 %                      |
| Accuracy (% of expected)       | 92.3 %                     |

\* true concentration relates to the original sample volume of 3 000 m<sup>3</sup>; see official text of the method for the details on spiking procedure

## 2,3,7,8-tetrachlorodibenzofuran

recovery from ambient air precision & recovery samples



Performance Summary Table

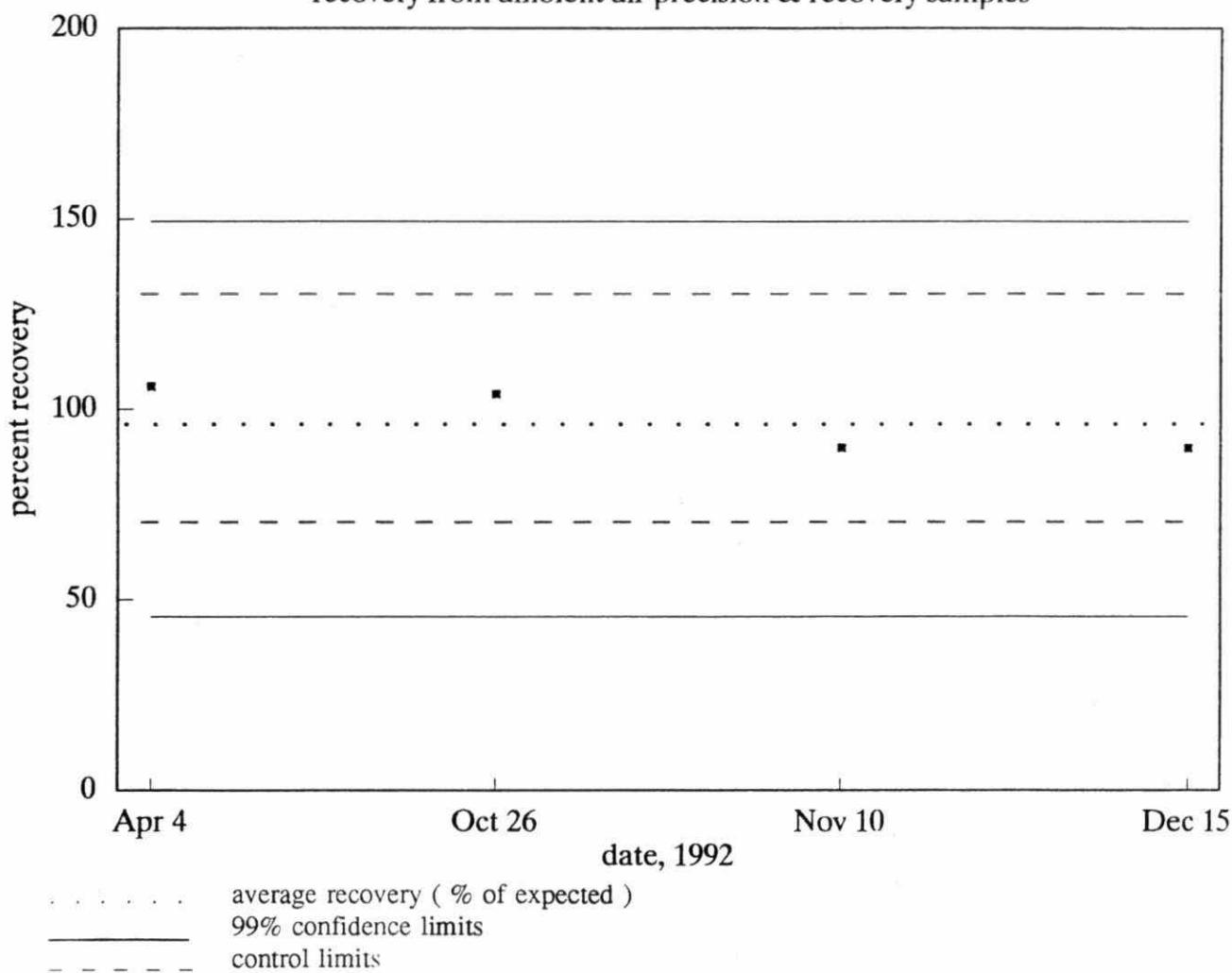
January - December 1992

|                                |                                 |
|--------------------------------|---------------------------------|
| Analyte                        | 2,3,7,8-tetrachlorodibenzofuran |
| True Concentration             | 0.27 pg/m <sup>3</sup> *        |
| Number of Observations         | 4                               |
| Between-run Standard Deviation | 5 %                             |
| Accuracy (% of expected)       | 94 %                            |

\* true concentration relates to the original sample volume of 3 000 m<sup>3</sup>; see official text of the method for the details on spiking procedure

## 2,3,4,7,8-pentachlorodibenzofuran

recovery from ambient air precision & recovery samples



Performance Summary Table

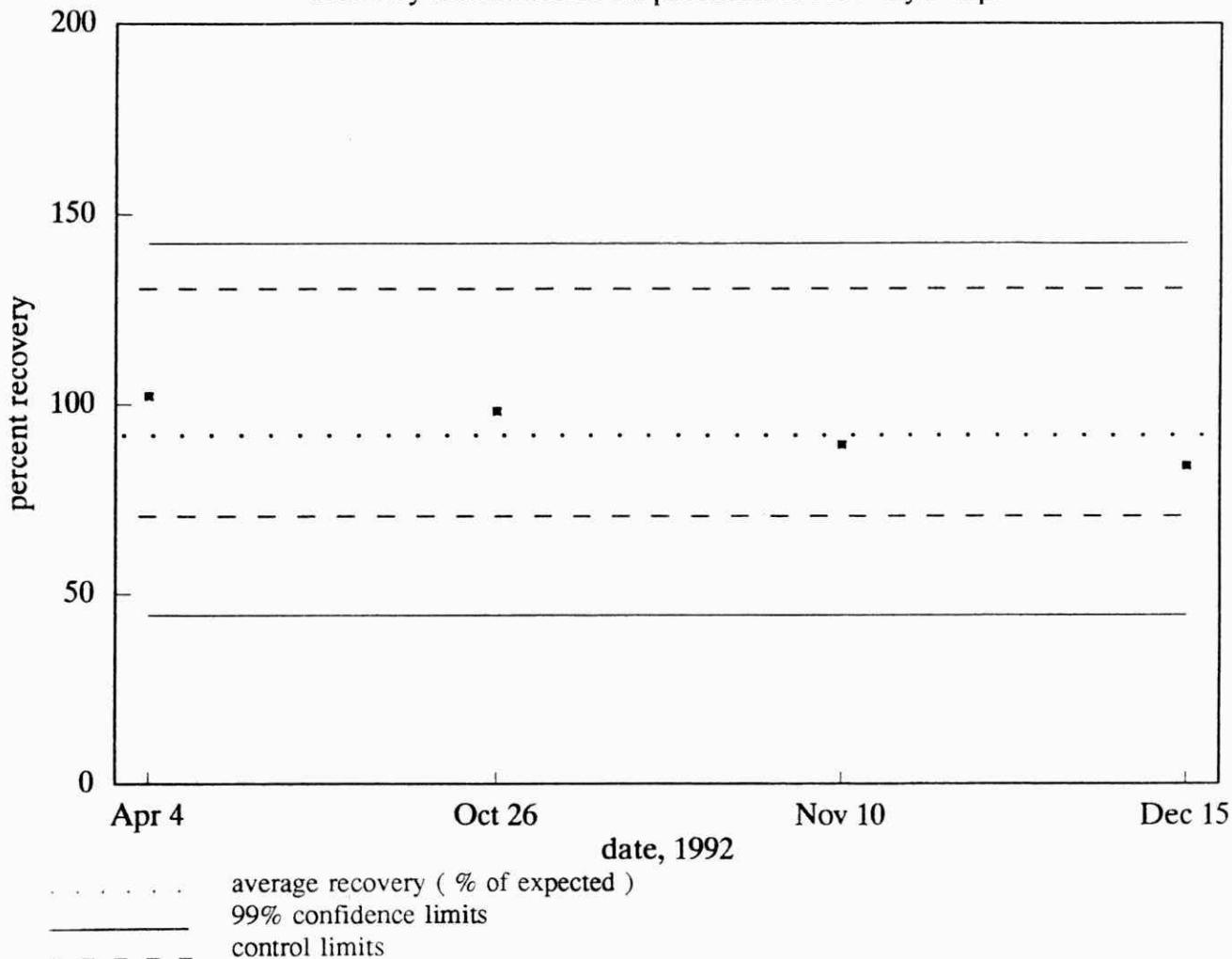
January - December 1992

|                                |                                   |
|--------------------------------|-----------------------------------|
| Analyte                        | 2,3,4,7,8-pentachlorodibenzofuran |
| True Concentration             | 1.3 pg/m <sup>3</sup> *           |
| Number of Observations         | 4                                 |
| Between-run Standard Deviation | 9 %                               |
| Accuracy (% of expected)       | 97 %                              |

\* true concentration relates to the original sample volume of 3 000 m<sup>3</sup>; see official text of the method for the details on spiking procedure

## 1,2,3,7,8-pentachlorodibenzofuran

recovery from ambient air precision & recovery samples



Performance Summary Table

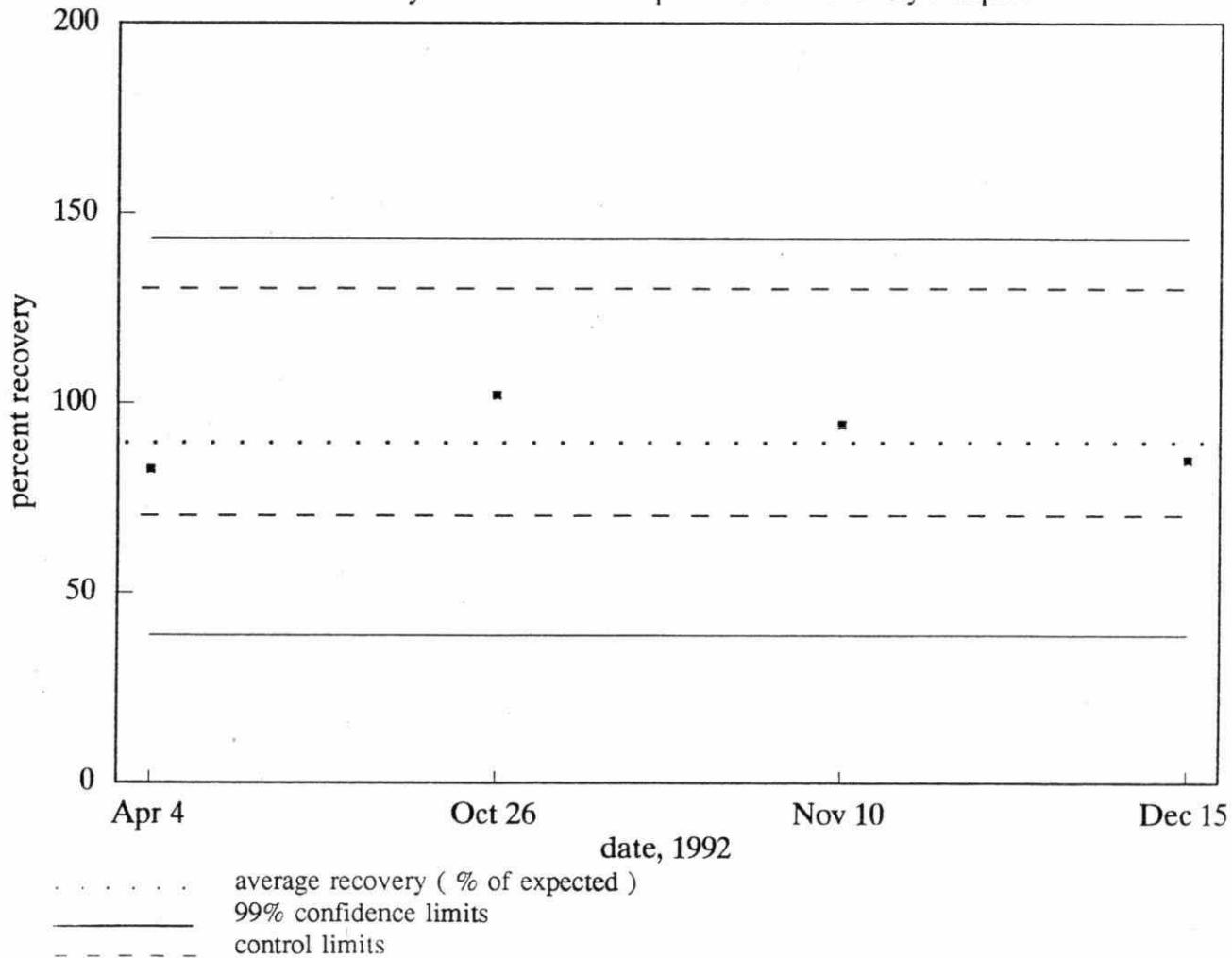
January - December 1992

|                                |                                   |
|--------------------------------|-----------------------------------|
| Analyte                        | 1,2,3,7,8-pentachlorodibenzofuran |
| True Concentration             | 1.3 pg/m <sup>3</sup>             |
| Number of Observations         | 4                                 |
| Between-run Standard Deviation | 8 %                               |
| Accuracy (% of expected)       | 93 %                              |

true concentration relates to the original sample volume of 3 000 m<sup>3</sup>; see official text of the method for the details on spiking procedure

## 1,2,3,4,7,8-hexachlorodibenzofuran

## recovery from ambient air precision & recovery samples



## Performance Summary Table

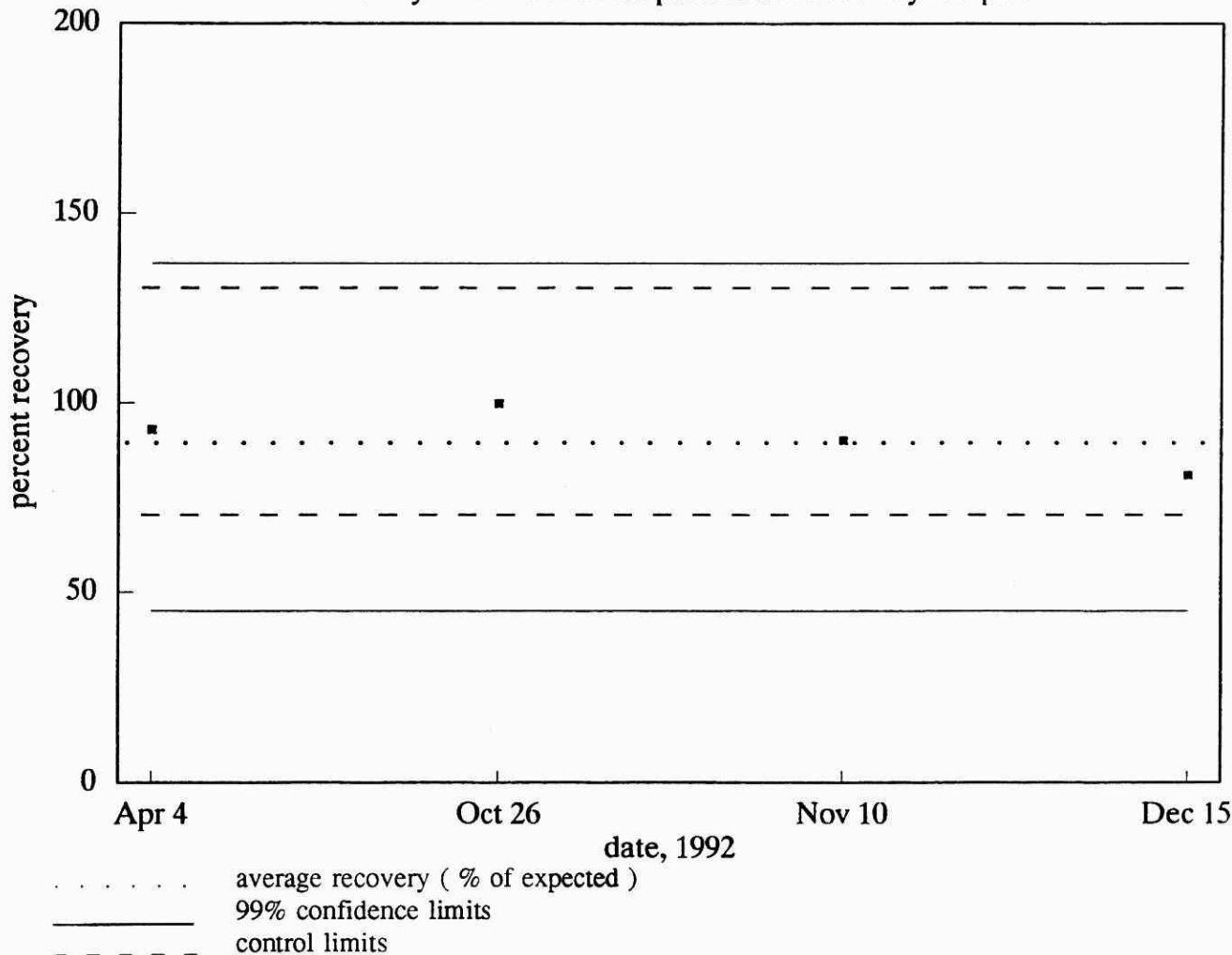
January - December 1992

|                                |                                    |
|--------------------------------|------------------------------------|
| Analyte                        | 1,2,3,4,7,8-hexachlorodibenzofuran |
| True Concentration             | 1.3 pg/m <sup>3</sup> *            |
| Number of Observations         | 4                                  |
| Between-run Standard Deviation | 9 %                                |
| Accuracy (% of expected)       | 91 %                               |

true concentration relates to the original sample volume of 3 000 m<sup>3</sup>; see official text of the method for the details on spiking procedure

## 1,2,3,6,7,8-hexachlorodibenzofuran

recovery from ambient air precision & recovery samples



Performance Summary Table

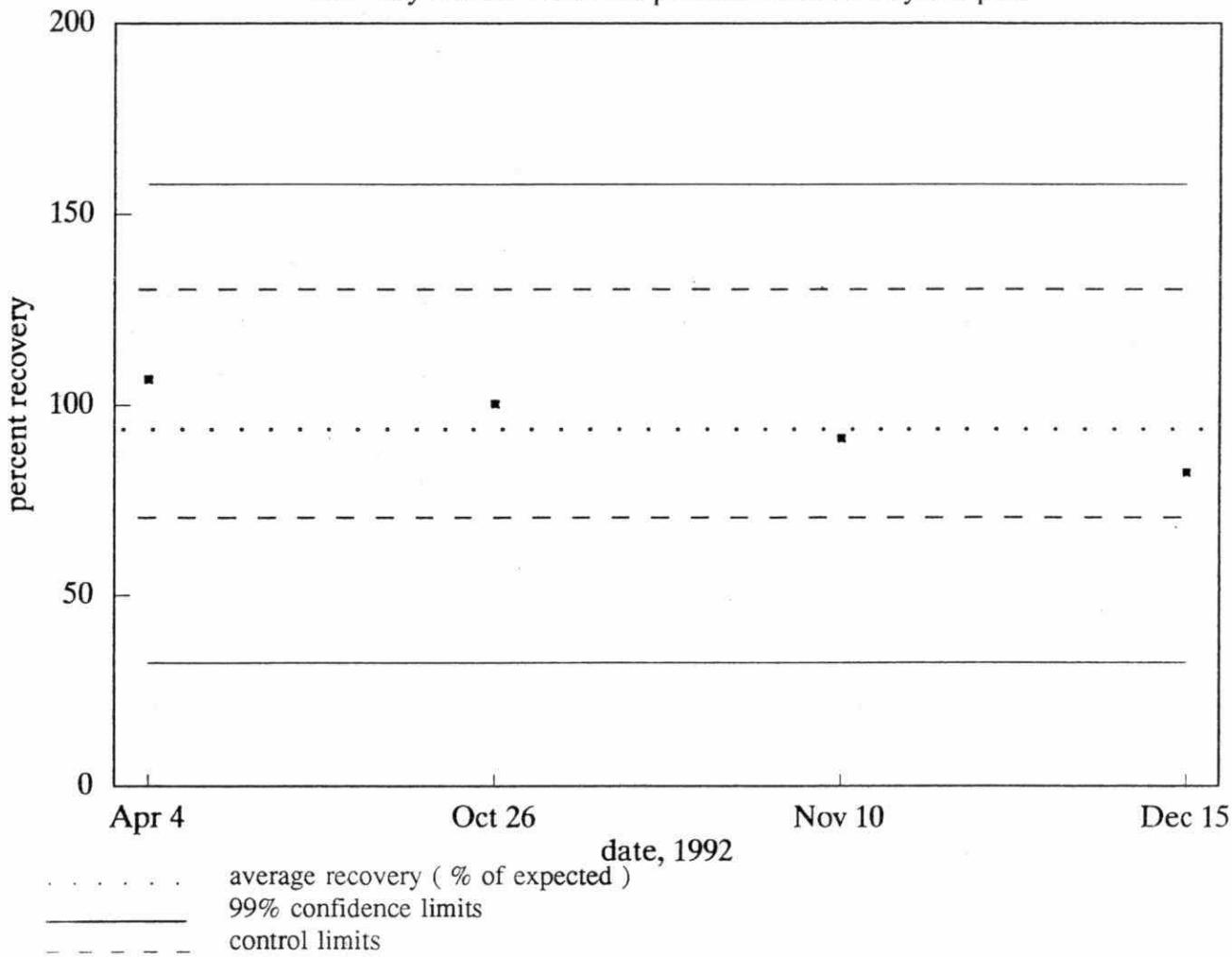
January - December 1992

|                                |                                    |
|--------------------------------|------------------------------------|
| Analyte                        | 1,2,3,6,7,8-hexachlorodibenzofuran |
| True Concentration             | 1.3 pg/m <sup>3</sup> *            |
| Number of Observations         | 4                                  |
| Between-run Standard Deviation | 8 %                                |
| Accuracy (% of expected)       | 91 %                               |

\* true concentration relates to the original sample volume of 3 000 m<sup>3</sup>; see official text of the method for the details on spiking procedure

## 2,3,4,6,7,8-hexachlorodibenzofuran

recovery from ambient air precision & recovery samples



## Performance Summary Table

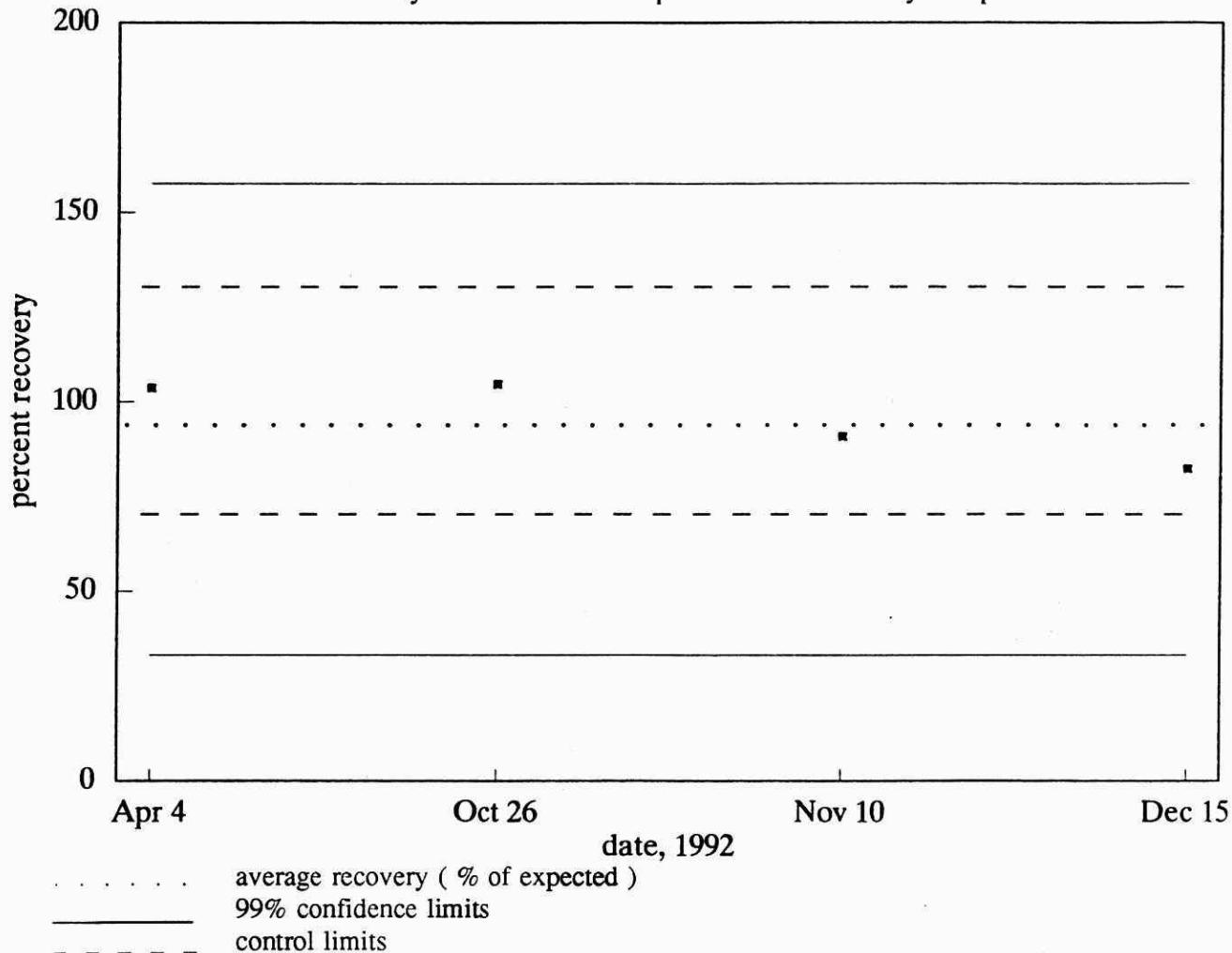
January - December 1992

|                                |                                    |
|--------------------------------|------------------------------------|
| Analyte                        | 2,3,4,6,7,8-hexachlorodibenzofuran |
| True Concentration             | 1.3 pg/m <sup>3</sup>              |
| Number of Observations         | 4                                  |
| Between-run Standard Deviation | 11 %                               |
| Accuracy (% of expected)       | 95 %                               |

true concentration relates to the original sample volume of 3 000 m<sup>3</sup>; see official text of the method for the details on spiking procedure

## 1,2,3,7,8,9-hexachlorodibenzofuran

recovery from ambient air precision & recovery samples



Performance Summary Table

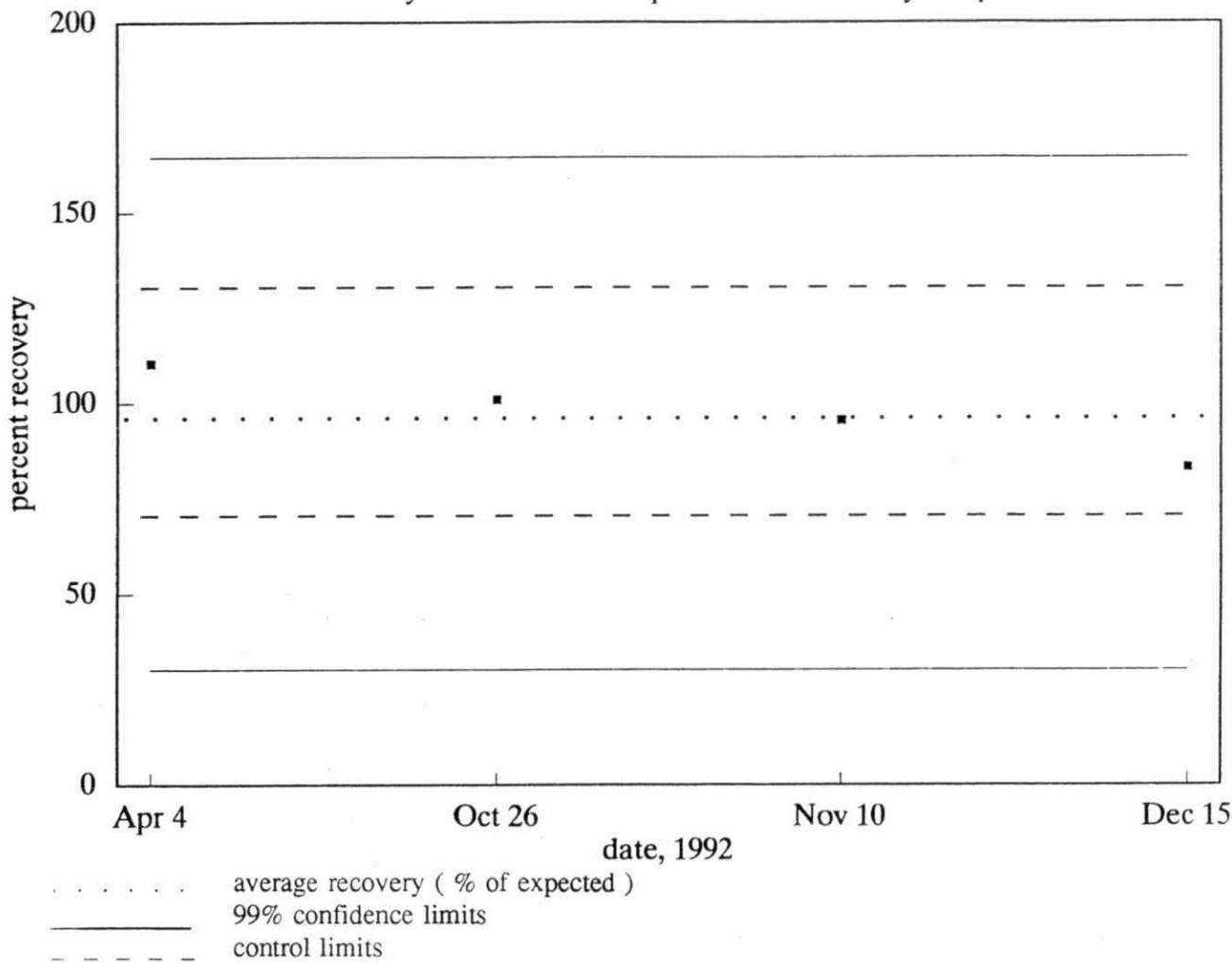
January - December 1992

|                                |                                    |
|--------------------------------|------------------------------------|
| Analyte                        | 1,2,3,7,8,9-hexachlorodibenzofuran |
| True Concentration             | 1.3 pg/m <sup>3</sup> *            |
| Number of Observations         | 4                                  |
| Between-run Standard Deviation | 11 %                               |
| Accuracy (% of expected)       | 95 %                               |

\* true concentration relates to the original sample volume of 3 000 m<sup>3</sup>; see official text of the method for the details on spiking procedure

## 1,2,3,4,6,7,8-heptachlorodibenzofuran

recovery from ambient air precision & recovery samples



Performance Summary Table

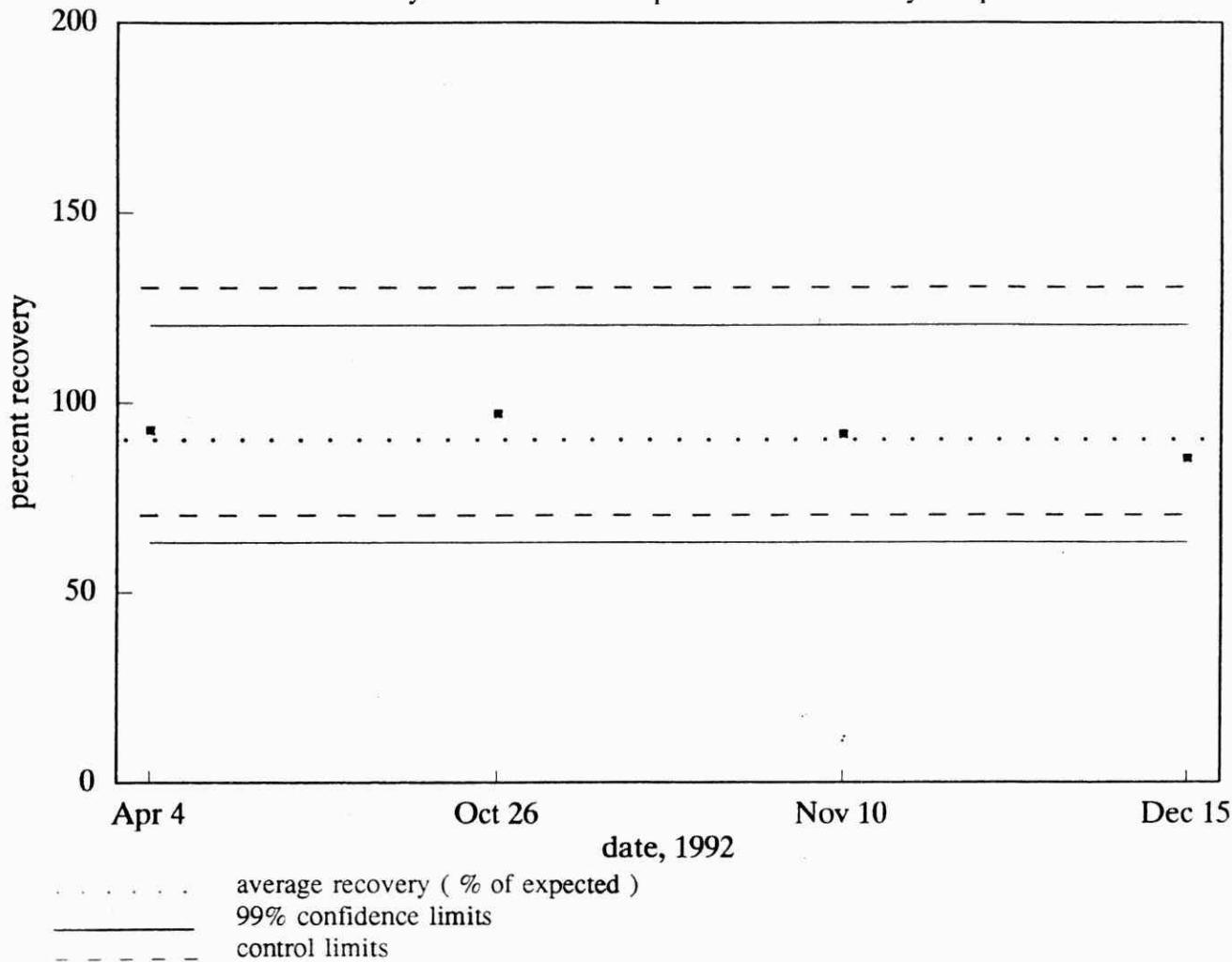
January - December 1992

|                                |                                       |
|--------------------------------|---------------------------------------|
| Analyte                        | 1,2,3,4,6,7,8-heptachlorodibenzofuran |
| True Concentration             | 1.3 pg/m <sup>3</sup> *               |
| Number of Observations         | 4                                     |
| Between-run Standard Deviation | 12 %                                  |
| Accuracy (% of expected)       | 97 %                                  |

\* true concentration relates to the original sample volume of 3 000 m<sup>3</sup>; see official text of the method for the details on spiking procedure

## 1,2,3,4,7,8,9-heptachlorodibenzofuran

recovery from ambient air precision & recovery samples



Performance Summary Table

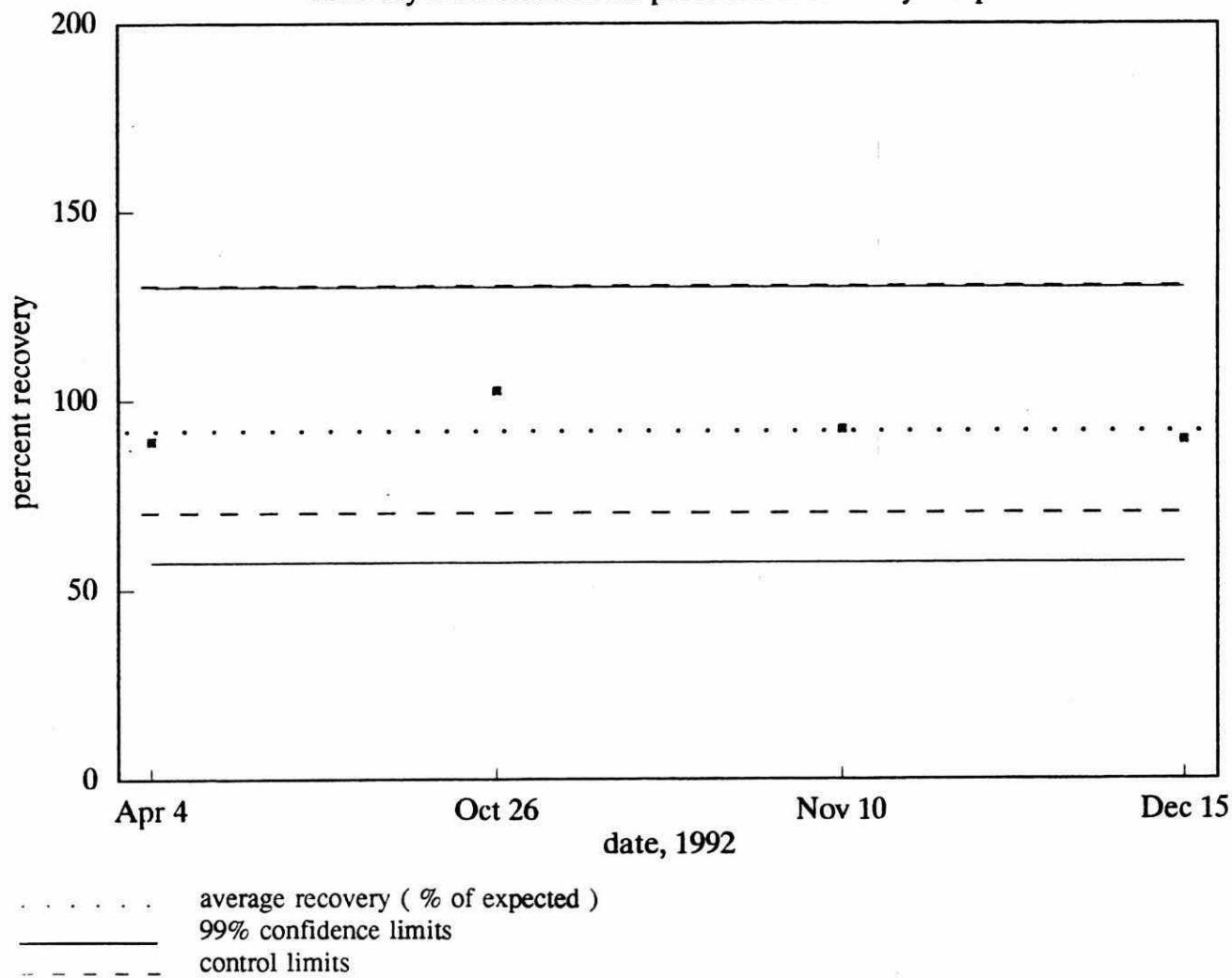
January - December 1992

|                                |                                       |
|--------------------------------|---------------------------------------|
| Analyte                        | 1,2,3,4,7,8,9-heptachlorodibenzofuran |
| True Concentration             | 1.3 pg/m <sup>3</sup> *               |
| Number of Observations         | 4                                     |
| Between-run Standard Deviation | 5 %                                   |
| Accuracy (% of expected)       | 92 %                                  |

true concentration relates to the original sample volume of 3 000 m<sup>3</sup>; see official text of the method for the details on spiking procedure

## octachlorodibenzofuran

recovery from ambient air precision & recovery samples



Performance Summary Table

January - December 1992

|                                |                        |
|--------------------------------|------------------------|
| Analyte                        | octachlorodibenzofuran |
| True Concentration             | 2.7 pg/m <sup>3</sup>  |
| Number of Observations         | 4                      |
| Between-run Standard Deviation | 6 %                    |
| Accuracy (% of expected)       | 94 %                   |

true concentration relates to the original sample volume of 3 000 m<sup>3</sup>; see official text of the method for the details on spiking procedure

**METHOD CODE :** PAAFD-E3318A

**METHOD TITLE:** The Determination of Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans in Precipitation by GC-MS

**LABORATORY :** Dioxin Unit

**SUPERVISOR :** Dr. E. Reiner

**SAMPLE TYPE :** precipitation ( aqueous samples containing less than 10% solids )

**PRINCIPLE OF THE METHOD :**

Samples consist of the separate portions: a cartridge containing XAD-2 Amberlite resin, a glass fibre filter and solvent used to rinse the collection funnel. A known quantity of isotopically labelled PCDDs and PCDFs is added to each sample to serve as an internal quantitation standard. The filter is solid/liquid extracted using a Soxhlet extractor and the XAD-2 resin is eluted with acetone/hexane. A multi-stage chromatographic cleanup procedure is used to remove potential chemical interferences.

The reconstituted final extract is examined by gas chromatography - high resolution mass spectrometry (GC-HRMS) or gas chromatography/tandem mass spectrometry (GC-MS-MS).

| <b>PARAMETERS MEASURED :</b>                       | <b>IDL ( pg )</b> |
|--|-------------------|
| 2,3,7,8-tetrachlorodibenzo-p-dioxin                | 5                 |
| 1,2,3,7,8-pentachlorodibenzo-p-dioxin              | 5                 |
| 1,2,3,4,7,8-hexachlorodibenzo-p-dioxin             | 10                |
| 1,2,3,6,7,8-hexachlorodibenzo-p-dioxin             | 10                |
| 1,2,3,7,8,9-hexachlorodibenzo-p-dioxin             | 10                |
| 1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin          | 15                |
| octachlorodibenzo-p-dioxin                         | 20                |
| 2,3,7,8-tetrachlorodibenzofuran                    | 5                 |
| 2,3,4,7,8-pentachlorodibenzofuran                  | 5                 |
| 1,2,3,7,8-pentachlorodibenzofuran                  | 5                 |
| 1,2,3,4,7,8-hexachlorodibenzofuran                 | 10                |
| 1,2,3,6,7,8-hexachlorodibenzofuran                 | 10                |
| 2,3,4,6,7,8-hexachlorodibenzofuran                 | 10                |
| 1,2,3,7,8,9-hexachlorodibenzofuran                 | 10                |
| 1,2,3,4,6,7,8-heptachlorodibenzofuran              | 15                |
| 1,2,3,4,7,8,9-heptachlorodibenzofuran              | 15                |
| octachlorodibenzofuran                             | 20                |
| total tetrachlorinated dibenzo-p-dioxins ( TCDD )  |                   |
| total pentachlorinated dibenzo-p-dioxins ( PCDD )  |                   |
| total hexachlorinated dibenzo-p-dioxins ( HxCDD )  |                   |
| total heptachlorinated dibenzo-p-dioxins ( HpCDD ) |                   |
| total tetrachlorinated dibenzofurans ( TCDF )      |                   |

( Parameters Measured continued )

total pentachlorinated dibenzofurans ( PCDF )  
total hexachlorinated dibenzofurans ( HxCDF )  
total heptachlorinated dibenzofurans ( HpCDF )

**REPORTING FORMAT :**

Results are reported in total picograms ( pg ) rounded off to 2 significant figures. The minimum reported levels are sample and analyte specific \* and range from 5 pg to 20 pg.

**QUALITY CONTROL :**

The routine quality control operations monitor overall method performance ( precision and recovery samples ), validity of calibration and consistency in injection volume ( injection standard ), absence of potential contamination ( method blanks ) and recovery of target analytes ( internal quantitation standard ).

**List of Performance Tables :**      Method Blanks Summary

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\* The minimum reported levels correspond to the amount of analyte that would give most-abundant ion response five times higher than corresponding instrumental noise.

Method Blanks Summary  
Precipitation - Filters

January 1992 - December 1992

| Analyte   | Number of Observations | Average Weight ( pg ) | Standard Deviation ( pg ) |
|---|------------------------|-----------------------|---------------------------|
| 2,3,7,8-tetrachlorodibenzo-p-dioxin                                     | 6                      | ND ( 5 )              |                           |
| 1,2,3,7,8-pentachlorodibenzo-p-dioxin                                   | 6                      | ND ( 5 )              |                           |
| 1,2,3,4,7,8-hexachlorodibenzo-p-dioxin                                  | 6                      | ND ( 10 )             |                           |
| 1,2,3,6,7,8-hexachlorodibenzo-p-dioxin                                  | 6                      | ND ( 10 )             |                           |
| 1,2,3,7,8,9-hexachlorodibenzo-p-dioxin                                  | 6                      | ND ( 10 )             |                           |
| 1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin<br>octachlorodibenzo-p-dioxin | 6                      | ND ( 15 )             |                           |
|   | 6                      | 2.7                   | 6.0                       |
| 2,3,7,8-tetrachlorodibenzofuran   | 6                      | ND ( 5 )              |                           |
| 2,3,4,7,8-pentachlorodibenzofuran                                       | 6                      | ND ( 5 )              |                           |
| 1,2,3,7,8-pentachlorodibenzofuran                                       | 6                      | ND ( 5 )              |                           |
| 1,2,3,4,7,8-hexachlorodibenzofuran                                      | 6                      | ND ( 10 )             |                           |
| 1,2,3,6,7,8-hexachlorodibenzofuran                                      | 6                      | ND ( 10 )             |                           |
| 2,3,4,6,7,8-hexachlorodibenzofuran                                      | 6                      | ND ( 10 )             |                           |
| 1,2,3,7,8,9-hexachlorodibenzofuran                                      | 6                      | ND ( 10 )             |                           |
| 1,2,3,4,6,7,8-heptachlorodibenzofuran                                   | 6                      | ND ( 15 )             |                           |
| 1,2,3,4,7,8,9-heptachlorodibenzofuran<br>octachlorodibenzofuran         | 6                      | ND ( 15 )             |                           |
|   | 6                      | ND ( 20 )             |                           |

ND ... Not detected. Detection limit in pg given in brackets ( ).



Ministry  
of the  
Environment

Ministère  
de  
l'Environnement

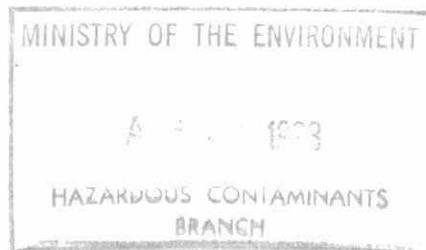
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## MEMORANDUM



DATE: August 16, 1993

TO: Distribution List

FROM: S. Villard  
Manager, Drinking Water Analyses Section

RE: DWO Section 1992 Performance Report

Enclosed please find a copy of the Drinking Water Organics Section 1992 Performance Report. This report summarizes the quality control procedures and the performance of the analytical methods used in the laboratories of the former Drinking Water Organics Section.

The Section has been renamed the Drinking Water Analyses Section. The DWA Section Performance Report for 1993 is planned to include the performance of the tests carried out in the Plasma Spectrometry Trace Metals Unit.

I would like to thank Bill Berg, Patrick Crozier, Eva Duchoslav, Eric Reiner and Vince Taguchi for the effort they put into realizing this report.

S. Villard

enclosure



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Method Blanks Summary  
Precipitation - XAD

January 1992 - December 1992

| Analyte                                   | Number of Observations | Average Weight ( pg ) | Standard Deviation ( pg ) |
|---|------------------------|-----------------------|---------------------------|
| 2,3,7,8-tetrachlorodibenzo-p-dioxin       | 6                      | ND ( 5 )              |                           |
| 1,2,3,7,8-pentachlorodibenzo-p-dioxin     | 6                      | ND ( 5 )              |                           |
| 1,2,3,4,7,8-hexachlorodibenzo-p-dioxin    | 6                      | ND ( 10 )             |                           |
| 1,2,3,6,7,8-hexachlorodibenzo-p-dioxin    | 6                      | ND ( 10 )             |                           |
| 1,2,3,7,8,9-hexachlorodibenzo-p-dioxin    | 6                      | ND ( 10 )             |                           |
| 1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin | 6                      | 2.2                   | 4.8                       |
| octachlorodibenzo-p-dioxin                | 6                      | 12                    | 20                        |
| 2,3,7,8-tetrachlorodibenzofuran           | 6                      | ND ( 5 )              |                           |
| 2,3,4,7,8-pentachlorodibenzofuran         | 6                      | ND ( 5 )              |                           |
| 1,2,3,7,8-pentachlorodibenzofuran         | 6                      | ND ( 5 )              |                           |
| 1,2,3,4,7,8-hexachlorodibenzofuran        | 6                      | ND ( 10 )             |                           |
| 1,2,3,6,7,8-hexachlorodibenzofuran        | 6                      | ND ( 10 )             |                           |
| 2,3,4,6,7,8-hexachlorodibenzofuran        | 6                      | ND ( 10 )             |                           |
| 1,2,3,7,8,9-hexachlorodibenzofuran        | 6                      | ND ( 10 )             |                           |
| 1,2,3,4,6,7,8-heptachlorodibenzofuran     | 6                      | ND ( 15 )             |                           |
| 1,2,3,4,7,8,9-heptachlorodibenzofuran     | 6                      | ND ( 15 )             |                           |
| octachlorodibenzofuran                    | 6                      | ND ( 20 )             |                           |

ND ... Not detected. Detection limits in pg given in brackets ( ).

Method Blanks Summary  
Precipitation - Funnel Rinses

January 1992 - December 1992

| Analyte                                   | Number of Observations | Average Weight ( pg ) | Standard Deviation ( pg ) |
|---|------------------------|-----------------------|---------------------------|
| 2,3,7,8-tetrachlorodibenzo-p-dioxin       | 5                      | ND ( 5 )              |                           |
| 1,2,3,7,8-pentachlorodibenzo-p-dioxin     | 5                      | ND ( 5 )              |                           |
| 1,2,3,4,7,8-hexachlorodibenzo-p-dioxin    | 5                      | ND ( 10 )             |                           |
| 1,2,3,6,7,8-hexachlorodibenzo-p-dioxin    | 5                      | ND ( 10 )             |                           |
| 1,2,3,7,8,9-hexachlorodibenzo-p-dioxin    | 5                      | ND ( 10 )             |                           |
| 1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin | 5                      | 11                    | 22                        |
| octachlorodibenzo-p-dioxin                | 5                      | 44                    | 88                        |
| 2,3,7,8-tetrachlorodibenzofuran           | 5                      | 3.4                   | 6.8                       |
| 2,3,4,7,8-pentachlorodibenzofuran         | 5                      | ND ( 5 )              |                           |
| 1,2,3,7,8-pentachlorodibenzofuran         | 5                      | 2.2                   | 4.4                       |
| 1,2,3,4,7,8-hexachlorodibenzofuran        | 5                      | ND ( 10 )             |                           |
| 1,2,3,6,7,8-hexachlorodibenzofuran        | 5                      | ND ( 10 )             |                           |
| 2,3,4,6,7,8-hexachlorodibenzofuran        | 5                      | ND ( 10 )             |                           |
| 1,2,3,7,8,9-hexachlorodibenzofuran        | 5                      | ND ( 10 )             |                           |
| 1,2,3,4,6,7,8-heptachlorodibenzofuran     | 5                      | 6                     | 12                        |
| 1,2,3,4,7,8,9-heptachlorodibenzofuran     | 5                      | ND ( 15 )             |                           |
| octachlorodibenzofuran                    | 5                      | 6                     | 12                        |

**METHOD CODE :** PFAFD-E3134A

**METHOD TITLE:** The Determination of Polychlorinated Dibeno-p-dioxins and Polychlorinated Dibenzofurans in Fish and Biota

**LABORATORY :** Dioxin Unit

**SUPERVISOR :** Dr. E. Reiner

**SAMPLE TYPE :** fish tissue and other biological tissue ( clams, shrimps )

**PRINCIPLE OF THE METHOD :**

Samples are homogenized by mechanical grinding of the tissue. A portion of the homogeneous sample is fortified with a known quantity of isotopically labelled PCDDs and PCDFs to serve as an internal quantitation standard and is digested overnight with concentrated hydrochloric acid. The digested solution is extracted with hexane and the extract is passed through a column containing anhydrous sodium sulphate and sulphuric acid-modified silica gel.

The extract is concentrated and subsequently fractionated using high performance liquid chromatography (HPLC). The reconstituted final extract is analyzed by gas chromatography - tandem mass spectrometry (GC-MS-MS) or gas chromatography - high resolution mass spectrometry (GC-HRMS).

**PARAMETERS MEASURED :**

**IDL ( pg/g )**

|   |   |
|---|---|
| 2,3,7,8-tetrachlorodibenzo-p-dioxin               | 1 |
| 1,2,3,7,8-pentachlorodibenzo-p-dioxin             | 1 |
| 1,2,3,4,7,8-hexachlorodibenzo-p-dioxin            | 2 |
| 1,2,3,6,7,8-hexachlorodibenzo-p-dioxin            | 2 |
| 1,2,3,7,8,9-hexachlorodibenzo-p-dioxin            | 2 |
| 1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin         | 3 |
| octachlorodibenzo-p-dioxin                        | 5 |
| 2,3,7,8-tetrachlorodibenzofuran                   | 1 |
| 2,3,4,7,8-pentachlorodibenzofuran                 | 1 |
| 1,2,3,7,8-pentachlorodibenzofuran                 | 1 |
| 1,2,3,4,7,8-hexachlorodibenzofuran                | 2 |
| 1,2,3,6,7,8-hexachlorodibenzofuran                | 2 |
| 2,3,4,6,7,8-hexachlorodibenzofuran                | 2 |
| 1,2,3,7,8,9-hexachlorodibenzofuran                | 2 |
| 1,2,3,4,6,7,8-heptachlorodibenzofuran             | 3 |
| 1,2,3,4,7,8,9-heptachlorodibenzofuran             | 3 |
| octachlorodibenzofuran                            | 5 |
| total tetrachlorinated dibenzo-p-dioxins ( TCDD ) |   |
| total pentachlorinated dibenzo-p-dioxins ( PCDD ) |   |
| total hexachlorinated dibenzo-p-dioxins ( HxCDD ) |   |

( Parameters Measured continued )

total heptachlorinated dibenzo-p-dioxins ( HpCDD )  
total tetrachlorinated dibenzofurans ( TCDF )  
total pentachlorinated dibenzofurans ( PCDF )  
total hexachlorinated dibenzofurans ( HxCDF )  
total heptachlorinated dibenzofurans ( HpCDF )

**REPORTING FORMAT :**

Results are reported as ppt ( picograms of CDD/CDF per gram of wet fish tissue ) rounded off to 2 significant figures. The minimum reported levels are sample and analyte specific and range from 1 pg/g to 10 pg/g.

**QUALITY CONTROL :**

The routine quality control operations monitor overall method performance ( precision and recovery samples ), validity of calibration and consistency in injection volume ( injection standard ), absence of potential contamination ( method blanks ) and recovery of target analytes ( internal quantitation standard ).

**REMARKS :** Two types of performance limits are displayed on the performance charts. One set was statistically derived from the 1992 data; while the other ( established at recoveries of 70% and 130% ) was adopted by the Dioxin Unit as the method performance control limits.

List of Performance Charts and Tables:

Method Blanks Summary  
2,3,7,8-tetrachlorodibenzo-p-dioxin  
1,2,3,7,8-pentachlorodibenzo-p-dioxin  
1,2,3,4,7,8-hexachlorodibenzo-p-dioxin  
1,2,3,6,7,8-hexachlorodibenzo-p-dioxin  
1,2,3,7,8,9-hexachlorodibenzo-p-dioxin  
1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin  
octachlorodibenzo-p-dioxin  
2,3,7,8-tetrachlorodibenzofuran  
2,3,4,7,8-pentachlorodibenzofuran  
1,2,3,7,8-pentachlorodibenzofuran  
1,2,3,4,7,8-hexachlorodibenzofuran  
1,2,3,6,7,8-hexachlorodibenzofuran  
2,3,4,6,7,8-hexachlorodibenzofuran  
1,2,3,7,8,9-hexachlorodibenzofuran  
1,2,3,4,6,7,8-heptachlorodibenzofuran  
1,2,3,4,7,8,9-heptachlorodibenzofuran  
octachlorodibenzofuran

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The minimum reported levels correspond to the amount of analyte that would give most-abundant ion response five times higher than corresponding instrumental noise.

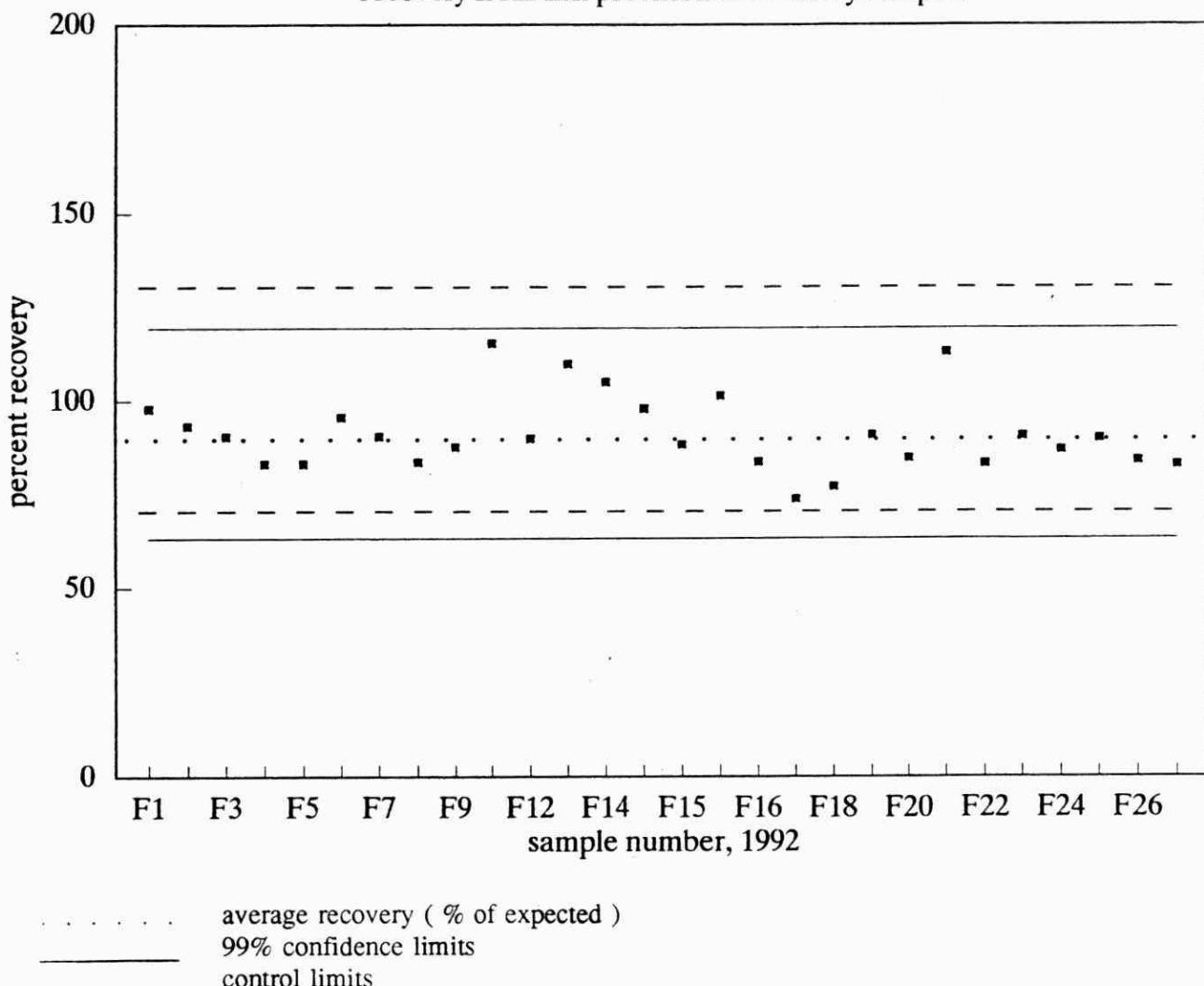
Method Blanks Summary

January 1992 - December 1992

| Analyte                                   | Number of Observations | Average Concentration ( pg/g ) | Standard Deviation ( pg/g ) |
|---|------------------------|--------------------------------|-----------------------------|
| 2,3,7,8-tetrachlorodibenzo-p-dioxin       | 21                     | 0.3                            | 1.1                         |
| 1,2,3,7,8-pentachlorodibenzo-p-dioxin     | 21                     | 1.1                            | 5.0                         |
| 1,2,3,4,7,8-hexachlorodibenzo-p-dioxin    | 21                     | 0.004                          | 0.020                       |
| 1,2,3,6,7,8-hexachlorodibenzo-p-dioxin    | 21                     | 0.007                          | 0.030                       |
| 1,2,3,7,8,9-hexachlorodibenzo-p-dioxin    | 21                     | 0.03                           | 0.12                        |
| 1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin | 21                     | 0.7                            | 3.3                         |
| octachlorodibenzo-p-dioxin                | 21                     | 5                              | 20                          |
| 2,3,7,8-tetrachlorodibenzofuran           | 21                     | 1.2                            | 3.1                         |
| 2,3,4,7,8-pentachlorodibenzofuran         | 21                     | 1.2                            | 5.5                         |
| 1,2,3,7,8-pentachlorodibenzofuran         | 21                     | 1.1                            | 4.9                         |
| 1,2,3,4,7,8-hexachlorodibenzofuran        | 21                     | 0.8                            | 3.0                         |
| 1,2,3,6,7,8-hexachlorodibenzofuran        | 21                     | 0.8                            | 3.3                         |
| 2,3,4,6,7,8-hexachlorodibenzofuran        | 21                     | 1.1                            | 4.5                         |
| 1,2,3,7,8,9-hexachlorodibenzofuran        | 21                     | 1.1                            | 4.4                         |
| 1,2,3,4,6,7,8-heptachlorodibenzofuran     | 21                     | 0.7                            | 2.8                         |
| 1,2,3,4,7,8,9-heptachlorodibenzofuran     | 21                     | 0.9                            | 3.2                         |
| octachlorodibenzofuran                    | 21                     | 2.3                            | 8.6                         |

## 2,3,7,8-tetrachlorodibenzo-p-dioxin

recovery from fish precision & recovery samples



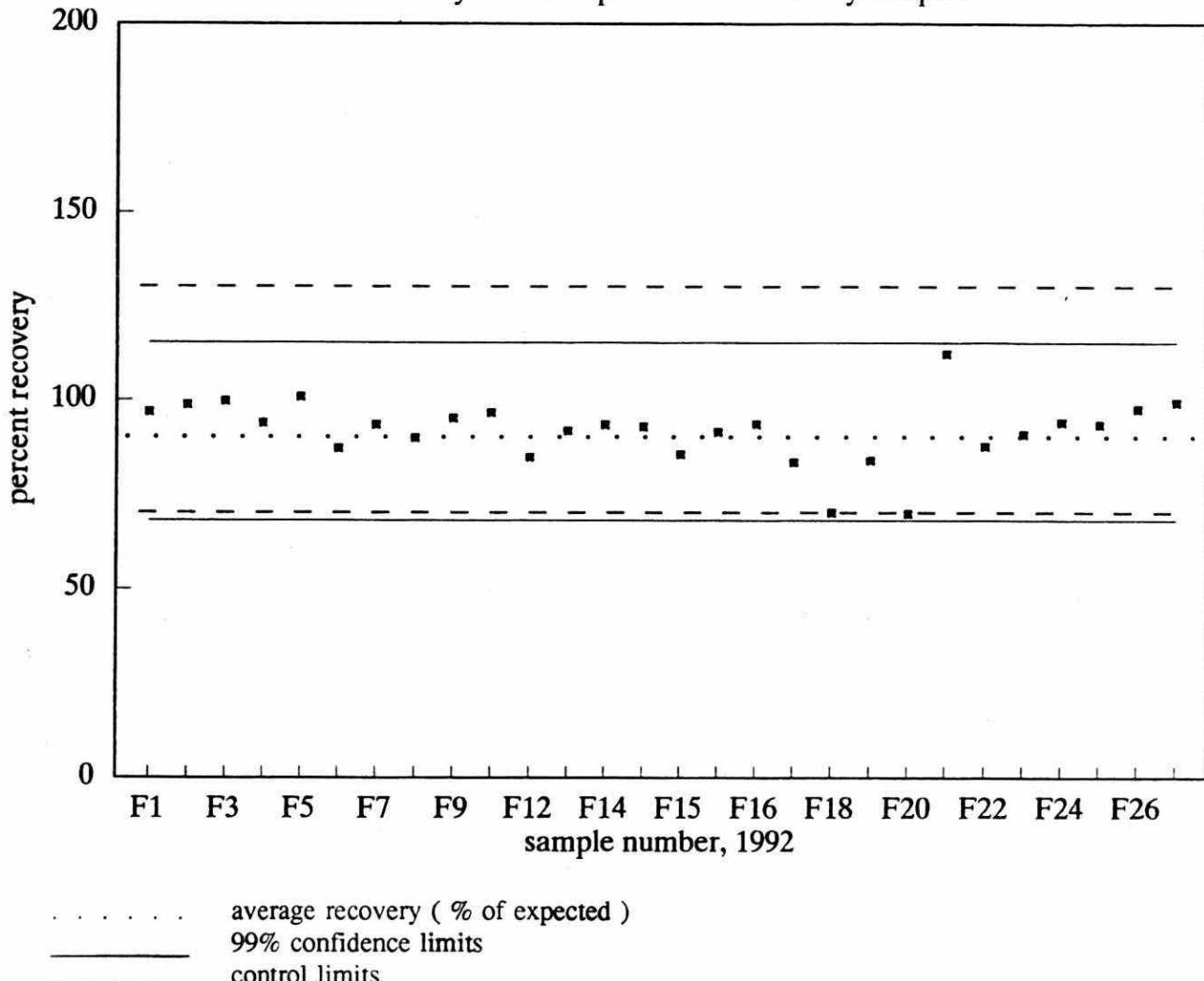
Performance Summary Table

January - December 1992

|                                |                                     |
|--------------------------------|-------------------------------------|
| Analyte                        | 2,3,7,8-tetrachlorodibenzo-p-dioxin |
| True Concentration             | 15 pg/g                             |
| Number of Observations         | 28                                  |
| Between-run Standard Deviation | 10 %                                |
| Accuracy (% of expected)       | 91 %                                |

## 1,2,3,7,8-pentachlorodibenzo-p-dioxin

recovery from fish precision & recovery samples



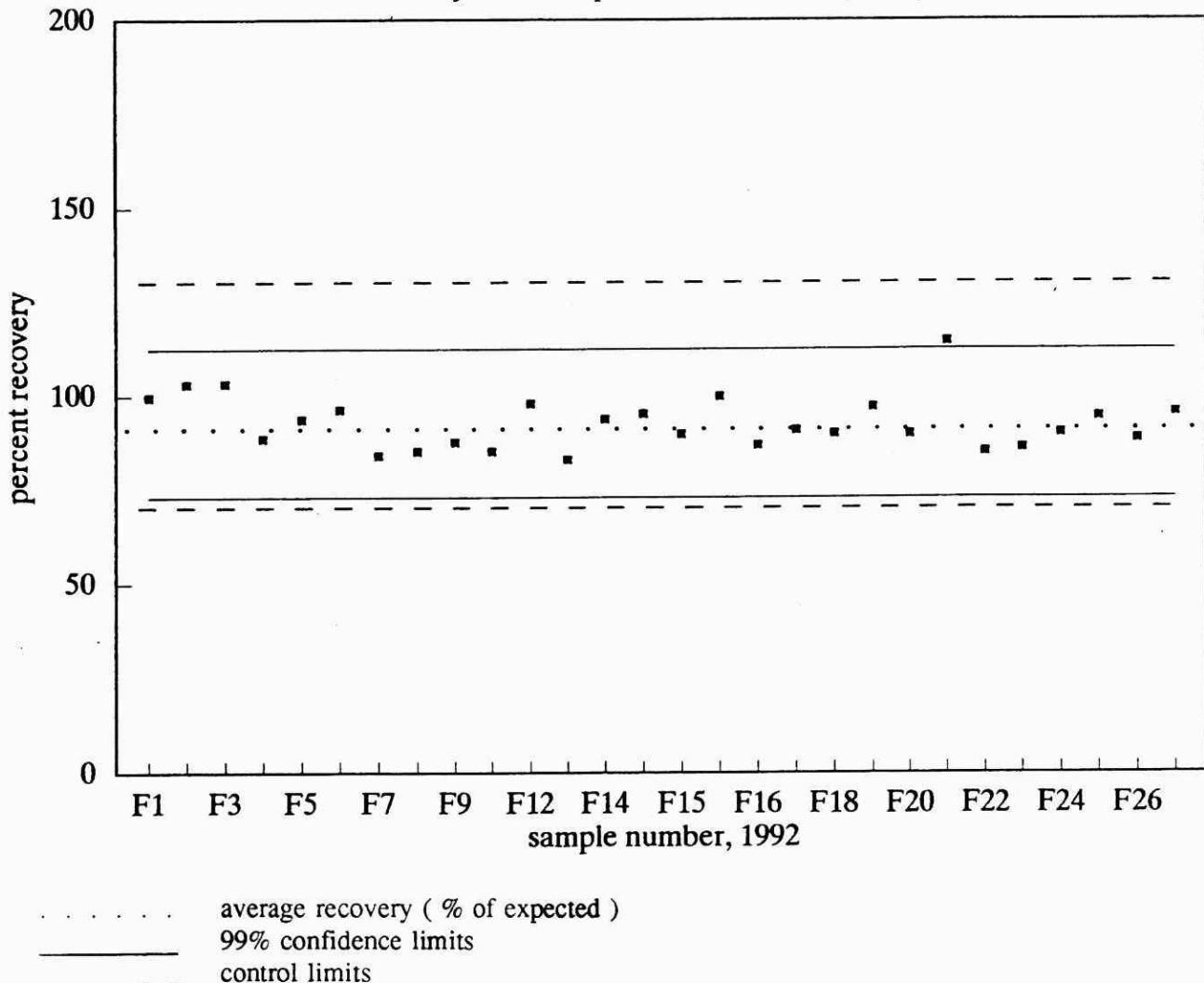
Performance Summary Table

January - December 1992

|                                |                                       |
|--------------------------------|---------------------------------------|
| Analyte                        | 1,2,3,7,8-pentachlorodibenzo-p-dioxin |
| True Concentration             | 75 pg/g                               |
| Number of Observations         | 28                                    |
| Between-run Standard Deviation | 9 %                                   |
| Accuracy (% of expected)       | 92 %                                  |

## 1,2,3,4,7,8-hexachlorodibenzo-p-dioxin

recovery from fish precision & recovery samples



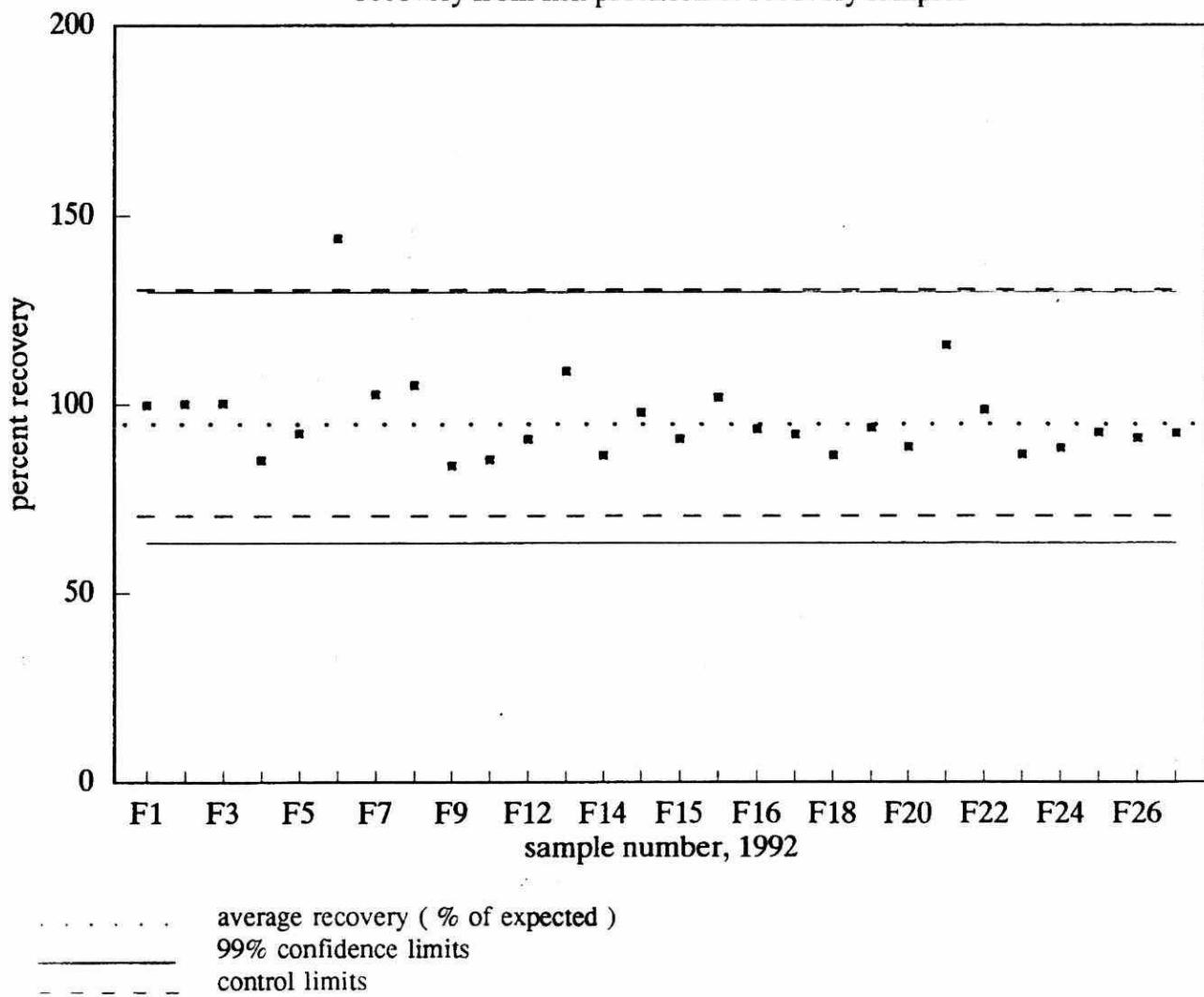
Performance Summary Table

January - December 1992

|                                |  |
|--------------------------------|--|
| Analyte                        | 1,2,3,4,7,8-hexachlorodibenzo-p-dioxin |
| True Concentration             | 75 pg/g                                |
| Number of Observations         | 28                                     |
| Between-run Standard Deviation | 7 %                                    |
| Accuracy (% of expected)       | 93 %                                   |

## 1,2,3,6,7,8-hexachlorodibenzo-p-dioxin

recovery from fish precision & recovery samples



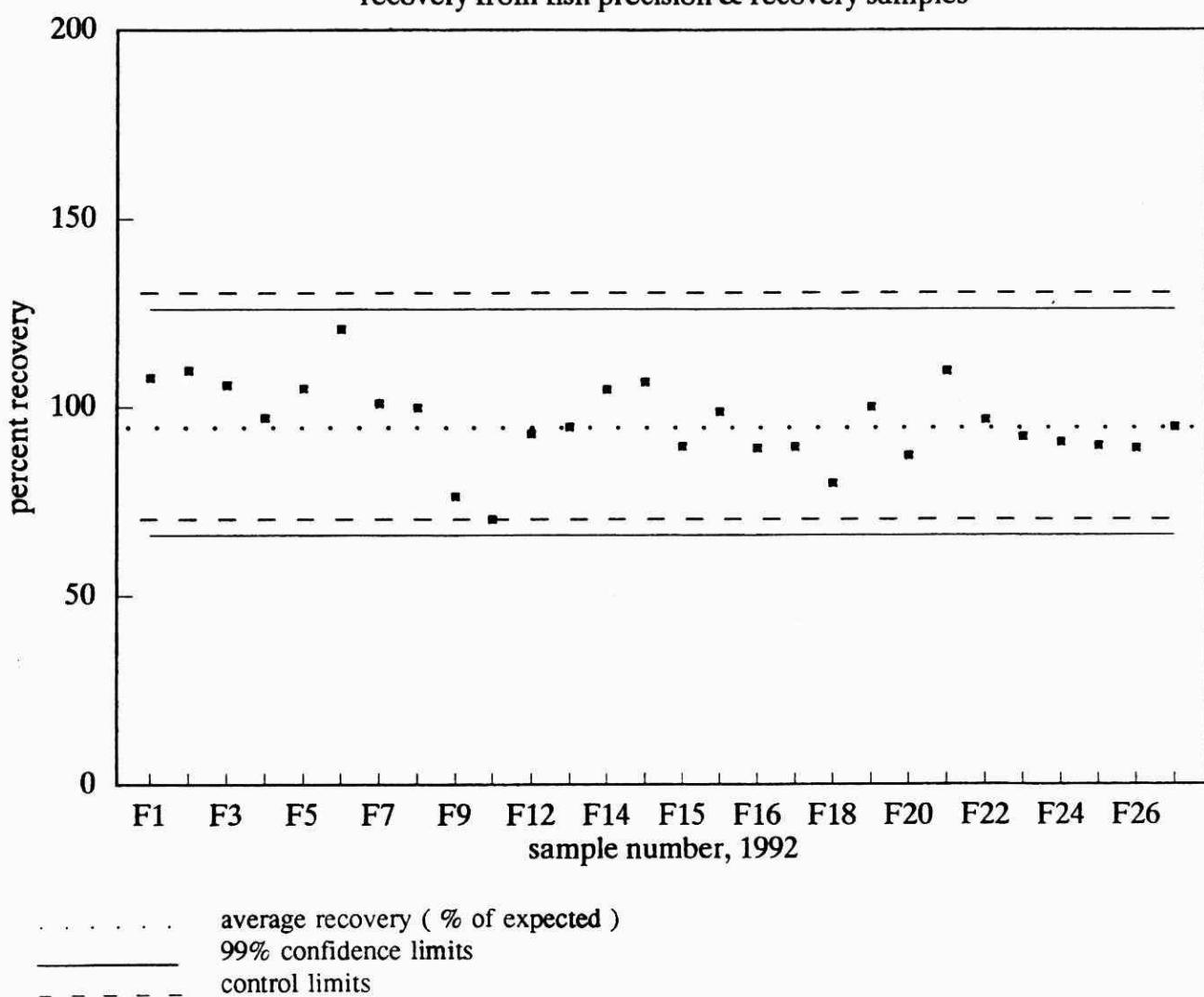
Performance Summary Table

January - December 1992

|                                |  |
|--------------------------------|--|
| Analyte                        | 1,2,3,6,7,8-hexachlorodibenzo-p-dioxin |
| True Concentration             | 75 pg/g                                |
| Number of Observations         | 26                                     |
| Between-run Standard Deviation | 12 %                                   |
| Accuracy (% of expected)       | 96 %                                   |

## 1,2,3,7,8,9-hexachlorodibenzo-p-dioxin

recovery from fish precision & recovery samples



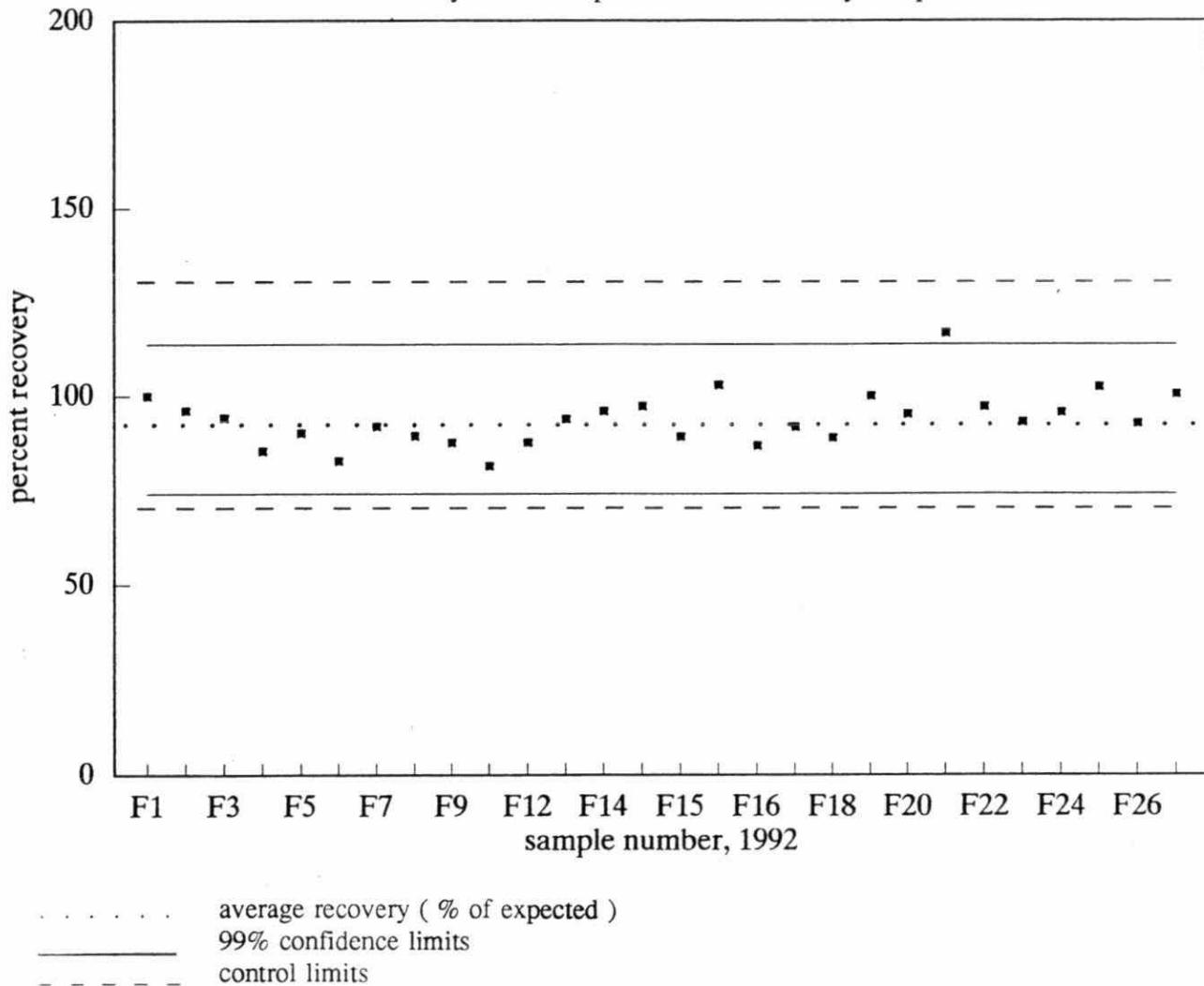
Performance Summary Table

January - December 1992

|                                |  |
|--------------------------------|--|
| Analyte                        | 1,2,3,7,8,9-hexachlorodibenzo-p-dioxin |
| True Concentration             | 75 pg/g                                |
| Number of Observations         | 26                                     |
| Between-run Standard Deviation | 11 %                                   |
| Accuracy (% of expected)       | 96 %                                   |

## 1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin

recovery from fish precision & recovery samples



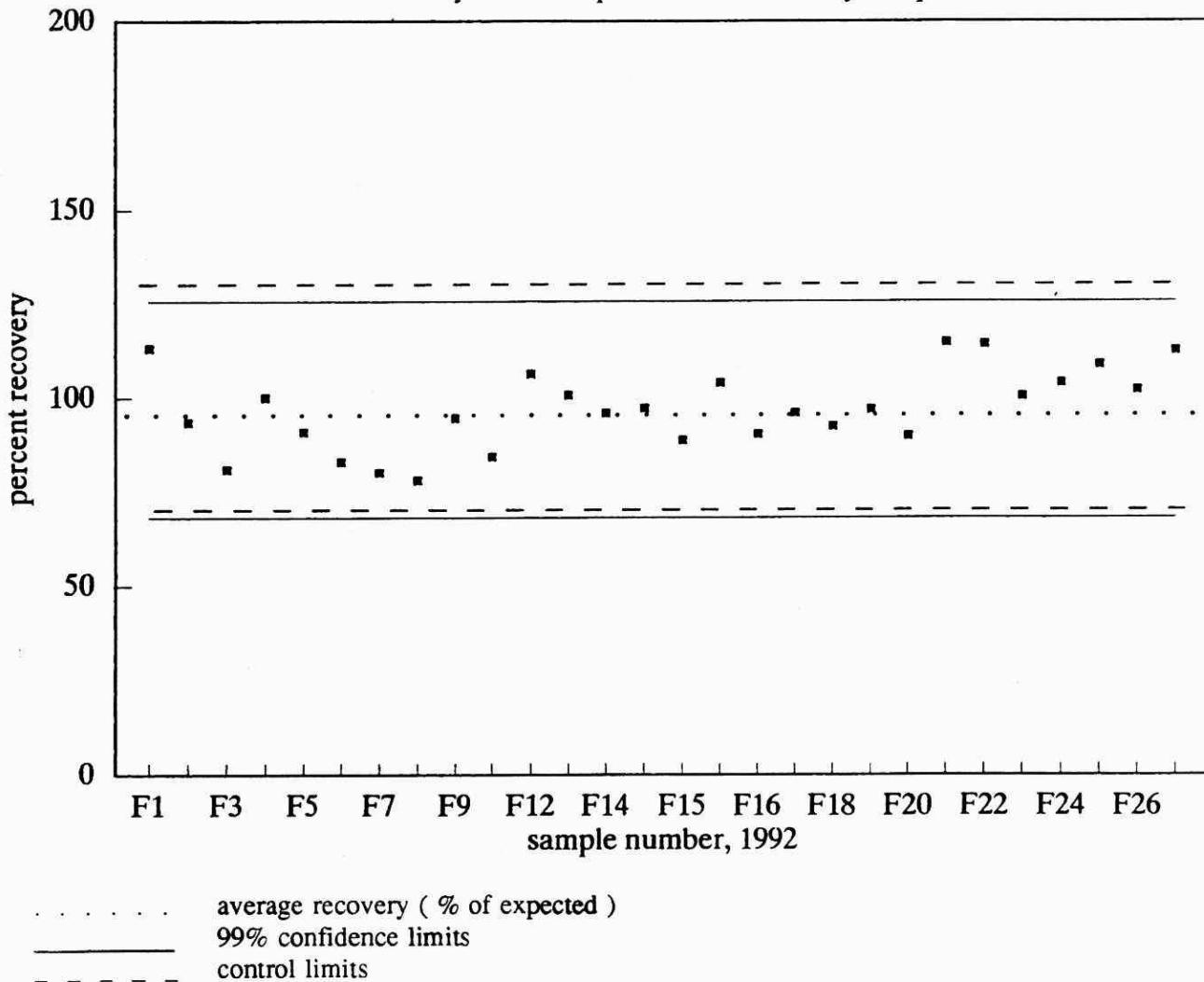
Performance Summary Table

January - December 1992

|                                |   |
|--------------------------------|---|
| Analyte                        | 1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin |
| True Concentration             | 75 pg/g                                   |
| Number of Observations         | 28  |
| Between-run Standard Deviation | 7 %                                       |
| Accuracy (% of expected)       | 94 %                                      |

## octachlorodibenzo-p-dioxin

recovery from fish precision & recovery samples



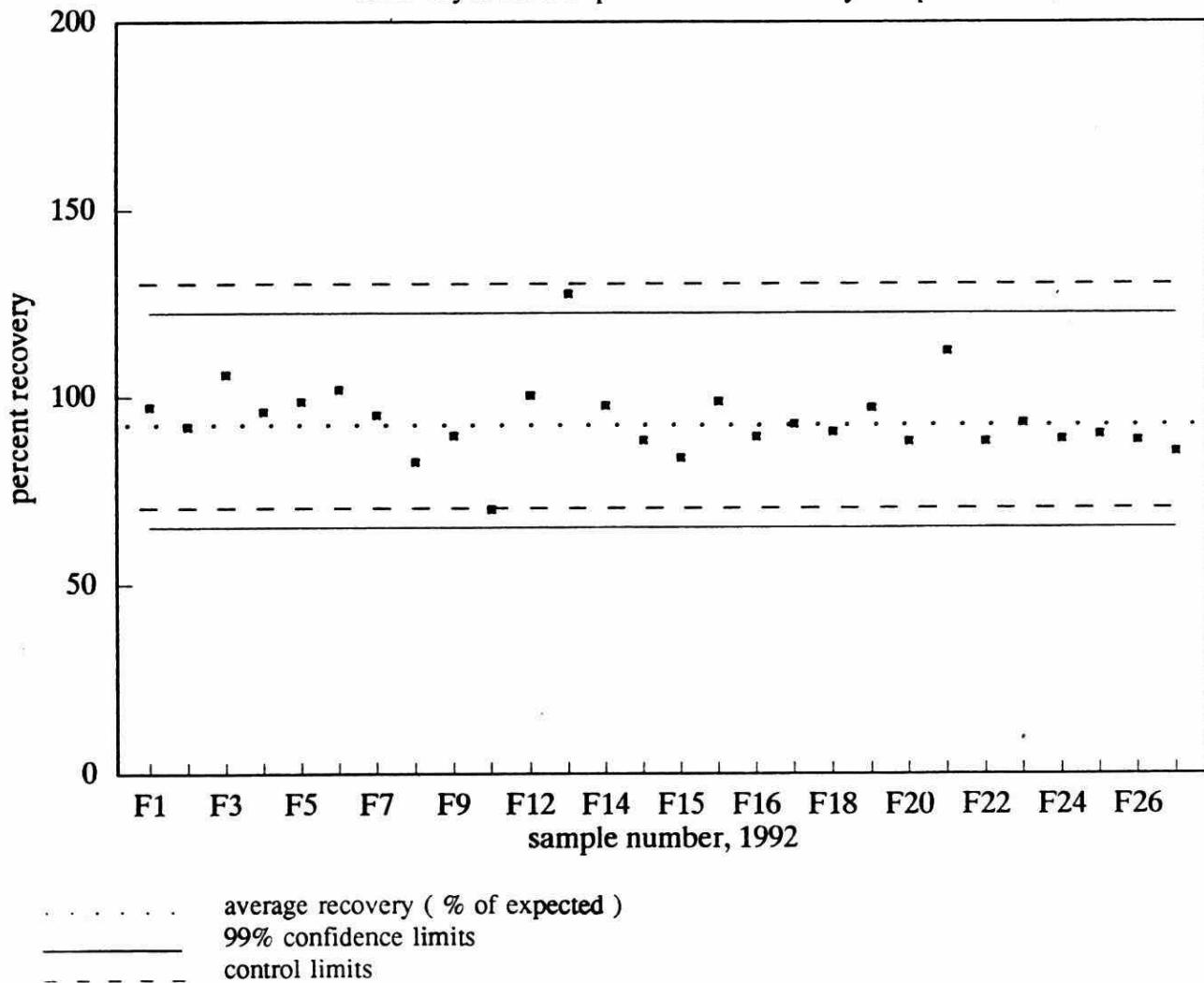
Performance Summary Table

January - December 1992

| Analyte                        | octachlorodibenzo-p-dioxin |
|--------------------------------|----------------------------|
| True Concentration             | 150 pg/g                   |
| Number of Observations         | 27                         |
| Between-run Standard Deviation | 10 %                       |
| Accuracy (% of expected)       | 97 %                       |

## 2,3,7,8-tetrachlorodibenzofuran

recovery from fish precision & recovery samples



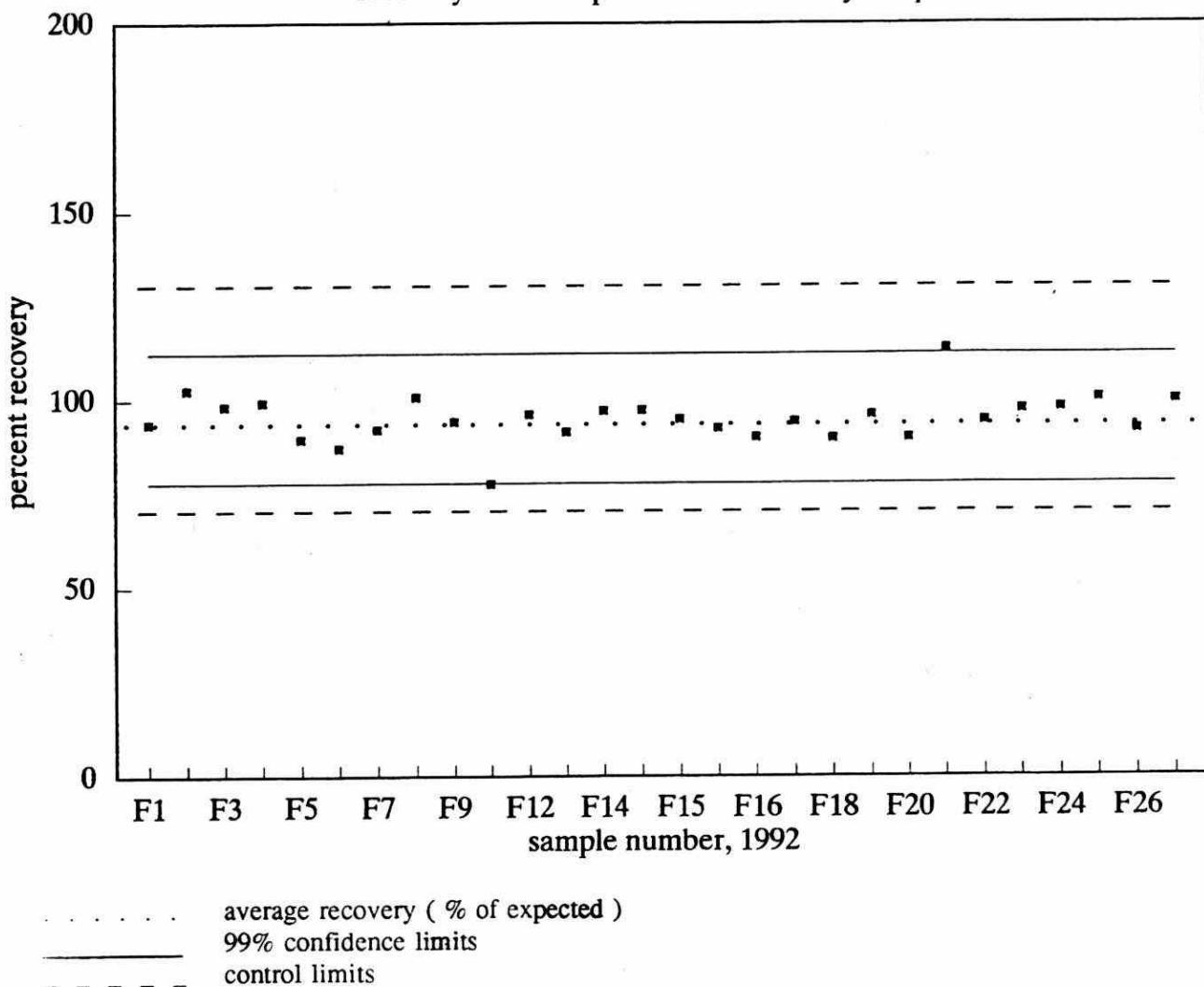
Performance Summary Table

January - December 1992

|                                |                                 |
|--------------------------------|---------------------------------|
| Analyte                        | 2,3,7,8-tetrachlorodibenzofuran |
| True Concentration             | 15 pg/g                         |
| Number of Observations         | 28                              |
| Between-run Standard Deviation | 10 %                            |
| Accuracy (% of expected)       | 94 %                            |

## 2,3,4,7,8-pentachlorodibenzofuran

recovery from fish precision & recovery samples



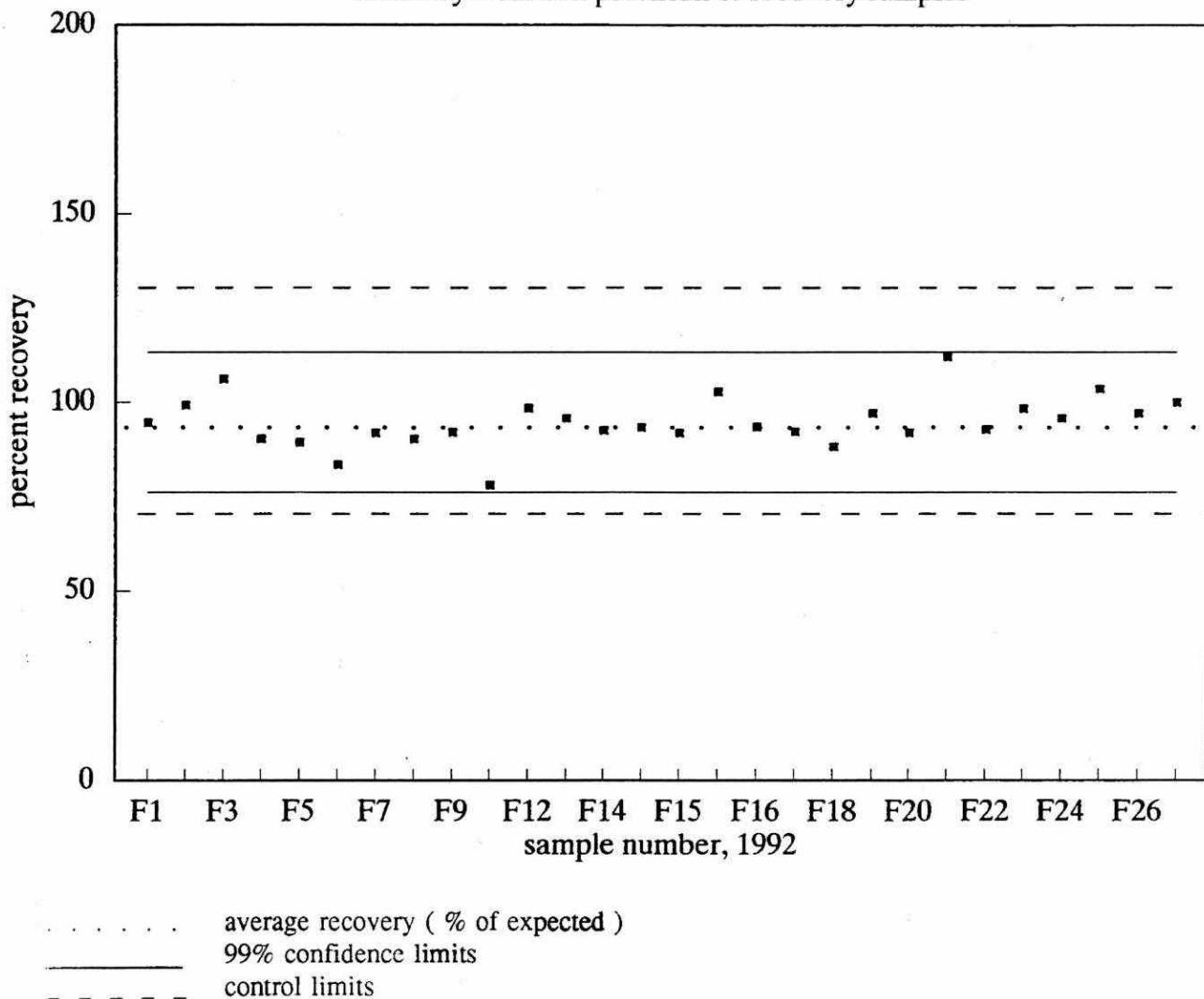
Performance Summary Table

January - December 1992

|                                |                                   |
|--------------------------------|-----------------------------------|
| Analyte                        | 2,3,4,7,8-pentachlorodibenzofuran |
| True Concentration             | 75 pg/g                           |
| Number of Observations         | 28                                |
| Between-run Standard Deviation | 6 %                               |
| Accuracy (% of expected)       | 95 %                              |

## 1,2,3,7,8-pentachlorodibenzofuran

recovery from fish precision & recovery samples



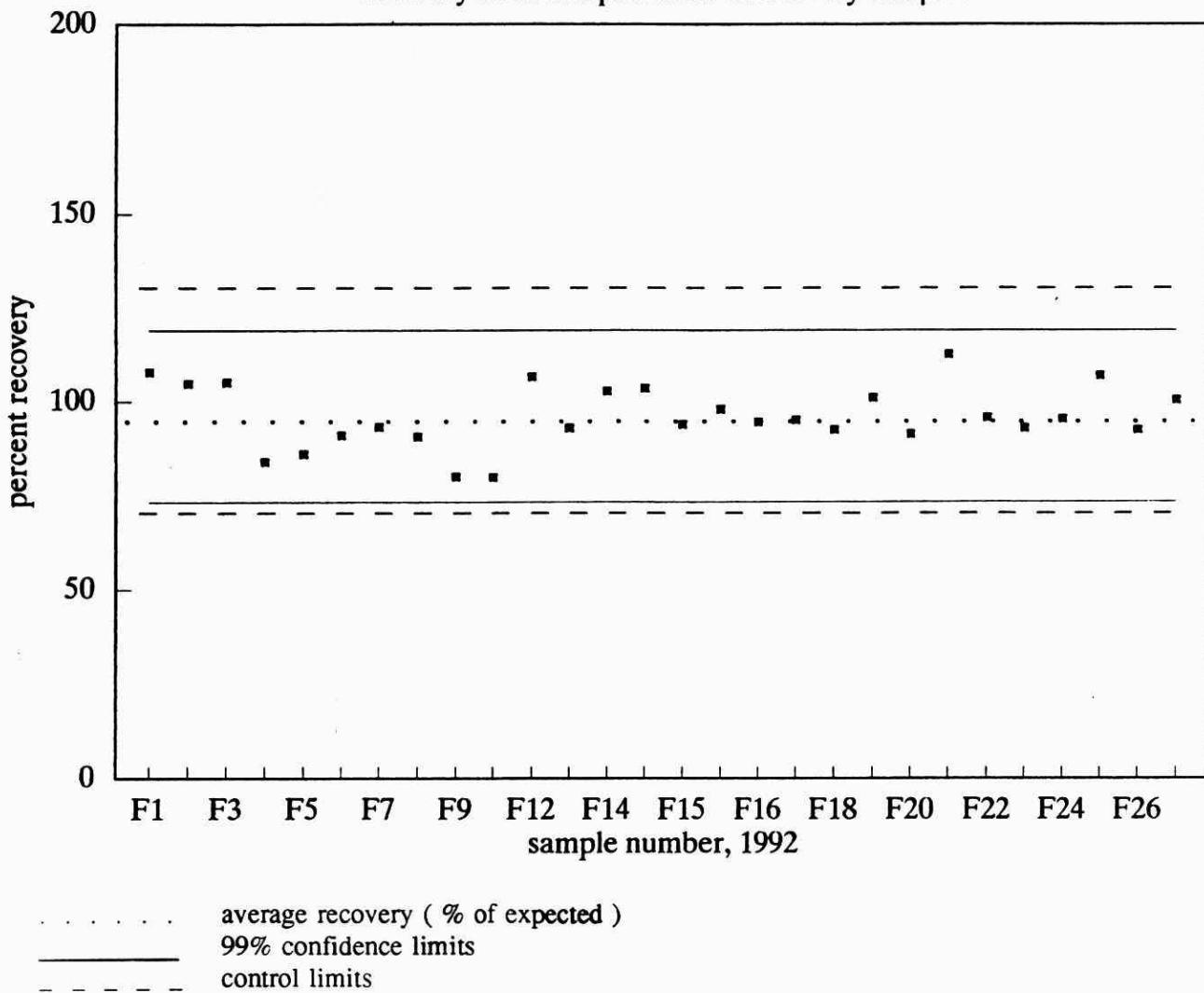
Performance Summary Table

January - December 1992

|                                |                                   |
|--------------------------------|-----------------------------------|
| Analyte                        | 1,2,3,7,8-pentachlorodibenzofuran |
| True Concentration             | 75 pg/g                           |
| Number of Observations         | 28                                |
| Between-run Standard Deviation | 7 %                               |
| Accuracy (% of expected)       | 95 %                              |

## 1,2,3,4,7,8-hexachlorodibenzofuran

recovery from fish precision & recovery samples



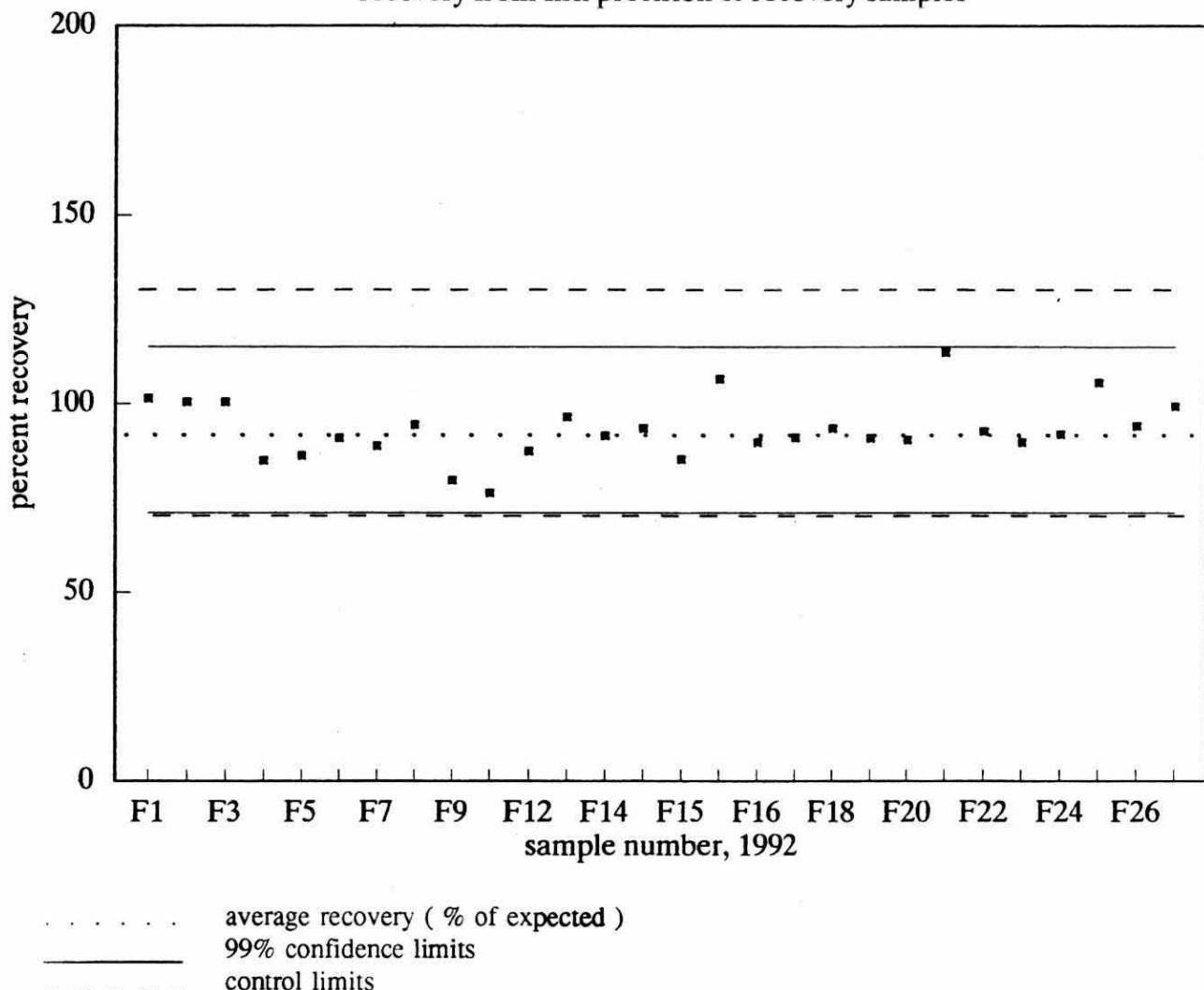
Performance Summary Table

January - December 1992

|                                |                                    |
|--------------------------------|------------------------------------|
| Analyte                        | 1,2,3,4,7,8-hexachlorodibenzofuran |
| True Concentration             | 75 pg/g                            |
| Number of Observations         | 28                                 |
| Between-run Standard Deviation | 8 %                                |
| Accuracy (% of expected)       | 96 %                               |

## 1,2,3,6,7,8-hexachlorodibenzofuran

recovery from fish precision & recovery samples



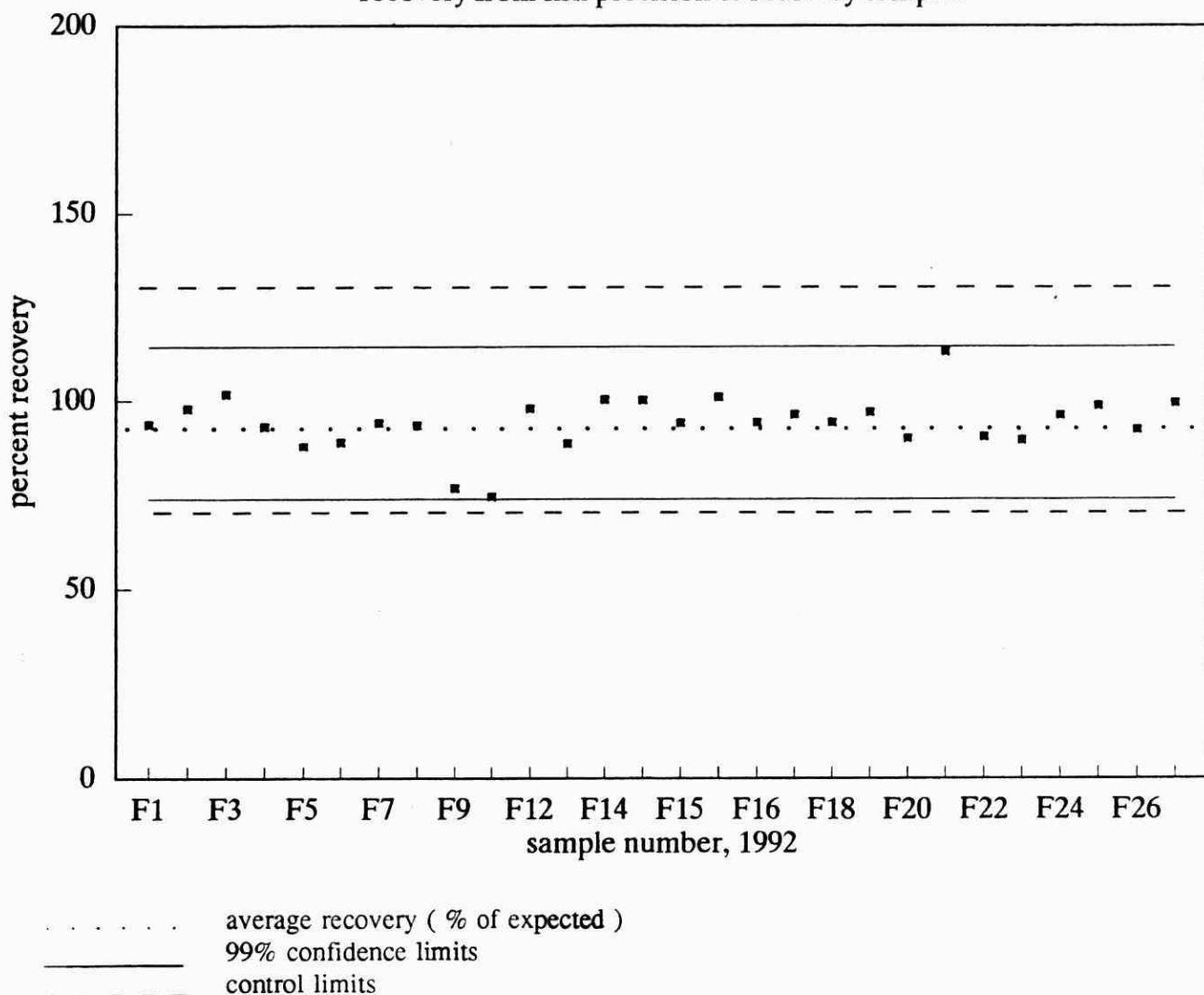
Performance Summary Table

January - December 1992

|                                |                                    |
|--------------------------------|------------------------------------|
| Analyte                        | 1,2,3,6,7,8-hexachlorodibenzofuran |
| True Concentration             | 75 pg/g                            |
| Number of Observations         | 28                                 |
| Between-run Standard Deviation | 8 %                                |
| Accuracy (% of expected)       | 93 %                               |

## 2,3,4,6,7,8-hexachlorodibenzofuran

recovery from fish precision & recovery samples



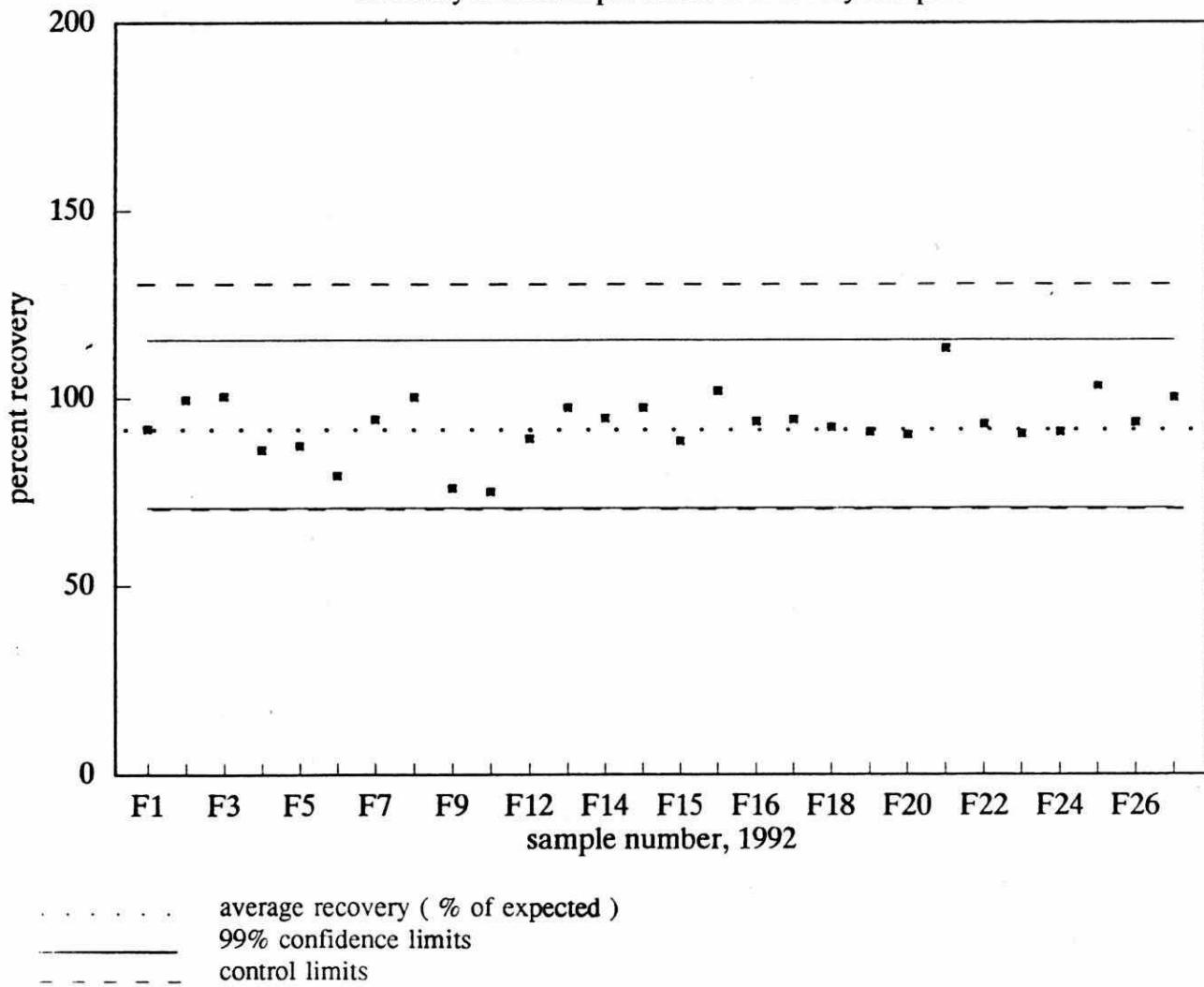
Performance Summary Table

January - December 1992

|                                |                                    |
|--------------------------------|------------------------------------|
| Analyte                        | 2,3,4,6,7,8-hexachlorodibenzofuran |
| True Concentration             | 75 pg/g                            |
| Number of Observations         | 28                                 |
| Between-run Standard Deviation | 7 %                                |
| Accuracy (% of expected)       | 94 %                               |

## 1,2,3,7,8,9-hexachlorodibenzofuran

recovery from fish precision & recovery samples



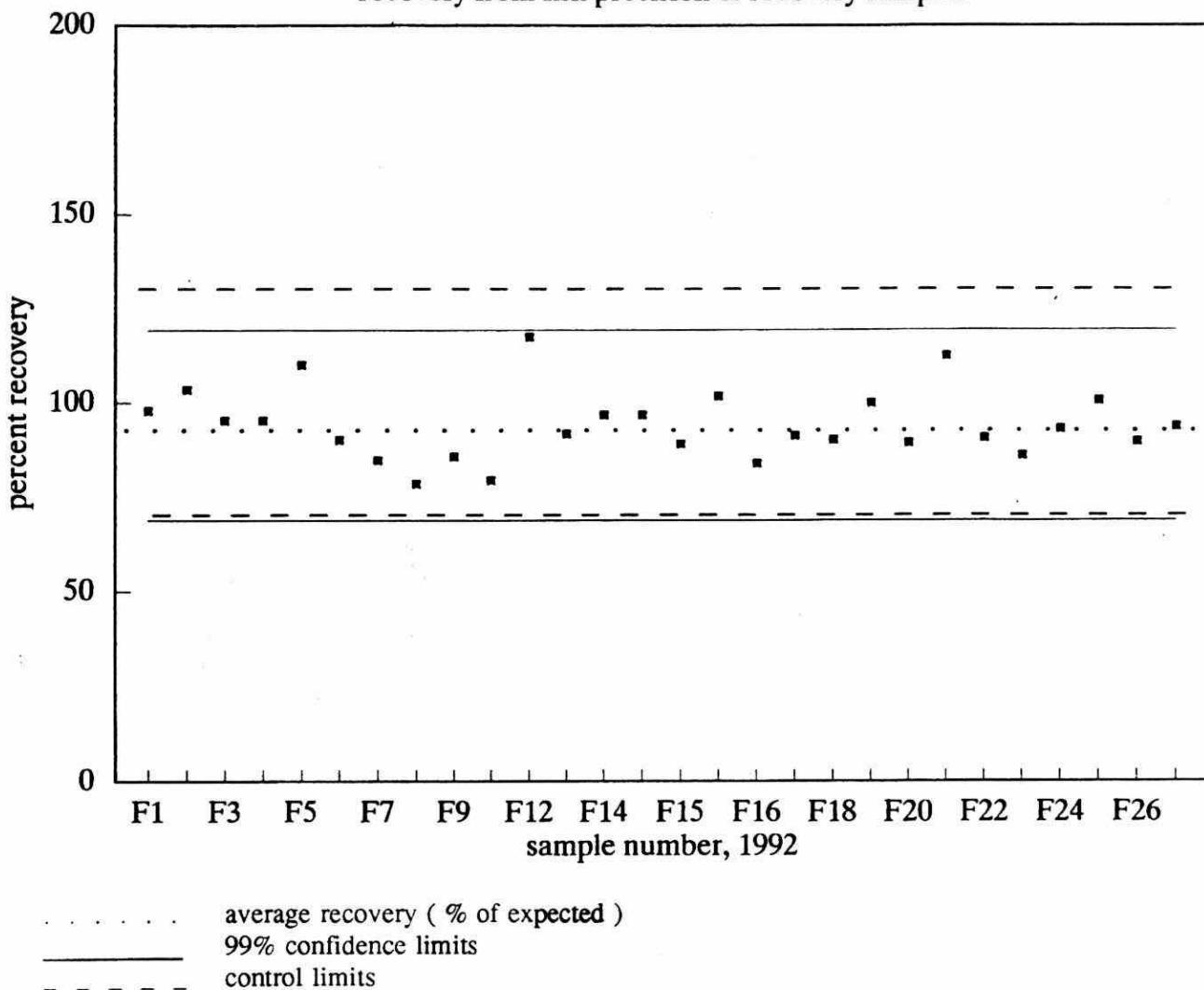
Performance Summary Table

January - December 1992

|                                |                                    |
|--------------------------------|------------------------------------|
| Analyte                        | 1,2,3,7,8,9-hexachlorodibenzofuran |
| True Concentration             | 75 pg/g                            |
| Number of Observations         | 28                                 |
| Between-run Standard Deviation | 8 %                                |
| Accuracy (% of expected)       | 93 %                               |

## 1,2,3,4,6,7,8-heptachlorodibenzofuran

recovery from fish precision & recovery samples



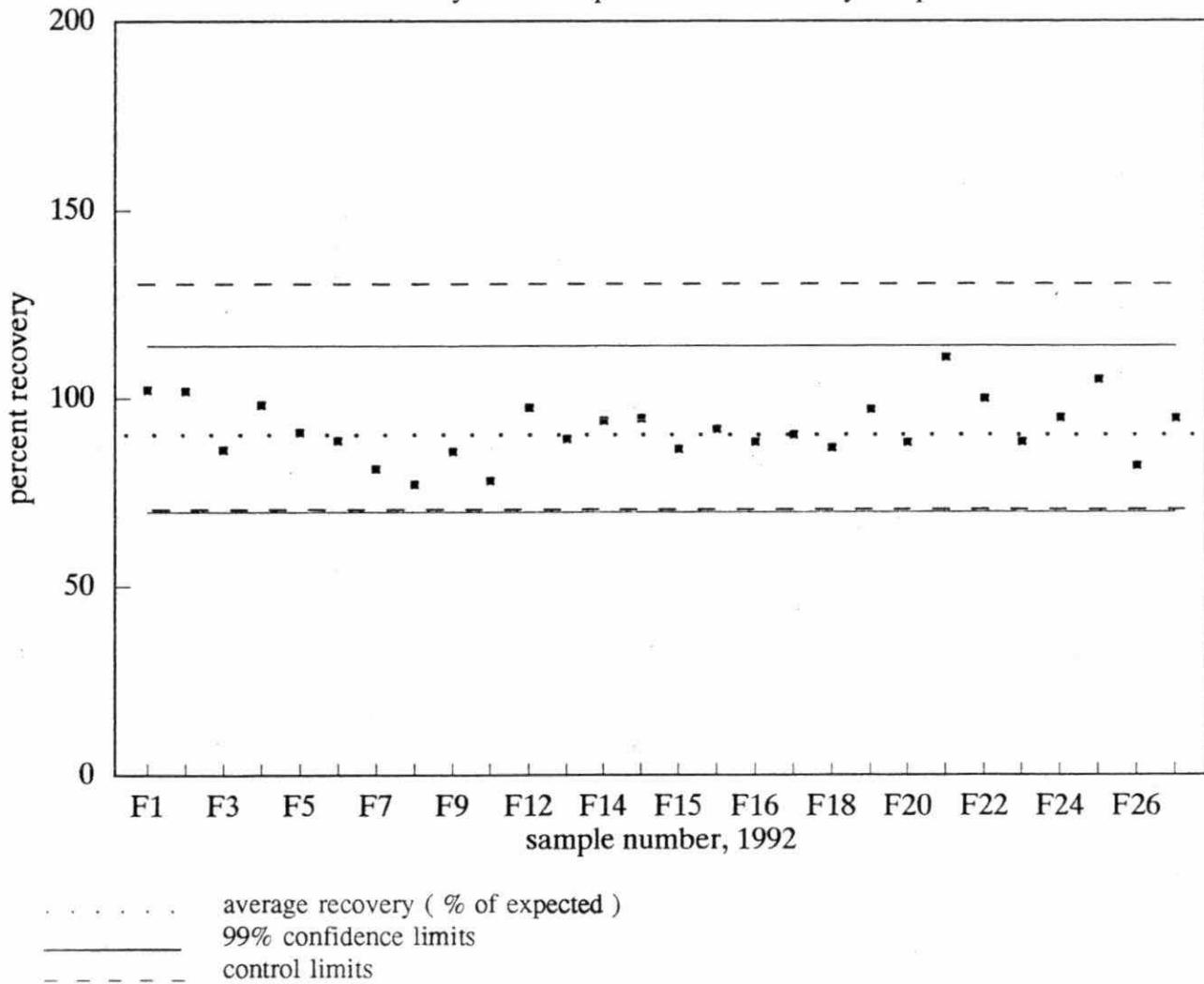
Performance Summary Table

January - December 1992

|                                |                                       |
|--------------------------------|---------------------------------------|
| Analyte                        | 1,2,3,4,6,7,8-heptachlorodibenzofuran |
| True Concentration             | 75 pg/g                               |
| Number of Observations         | 28                                    |
| Between-run Standard Deviation | 9 %                                   |
| Accuracy (% of expected)       | 94 %                                  |

## 1,2,3,4,7,8,9-heptachlorodibenzofuran

recovery from fish precision & recovery samples



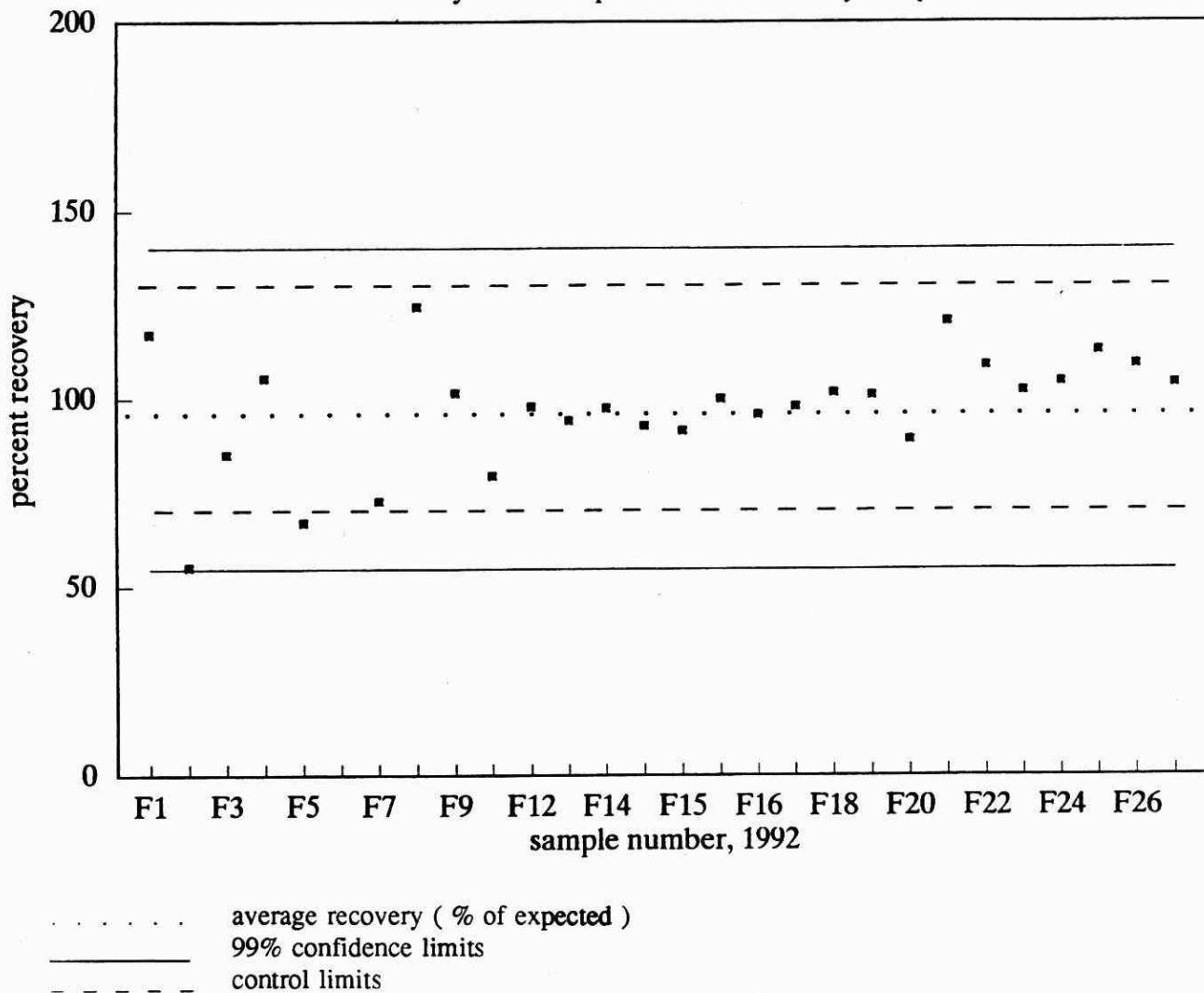
Performance Summary Table

January - December 1992

|                                |                                       |
|--------------------------------|---------------------------------------|
| Analyte                        | 1,2,3,4,7,8,9-heptachlorodibenzofuran |
| True Concentration             | 75 pg/g                               |
| Number of Observations         | 28                                    |
| Between-run Standard Deviation | 8 %                                   |
| Accuracy (% of expected)       | 92 %                                  |

## octachlorodibenzofuran

recovery from fish precision & recovery samples



Performance Summary Table

January - December 1992

| Analyte                        | octachlorodibenzofuran |
|--------------------------------|------------------------|
| True Concentration             | 150 pg/g               |
| Number of Observations         | 27                     |
| Between-run Standard Deviation | 16 %                   |
| Accuracy (% of expected)       | 97 %                   |

**METHOD CODE :** PSAFD-E3151B

**METHOD TITLE:** The Determination of Polychlorinated Dibenzo-p-dioxins (PCDD) and Polychlorinated Dibenzofurans (PCDF) in Soil and Sediment

**LABORATORY :** Dioxin Unit

**SUPERVISOR :** Dr. E. Reiner

**SAMPLE TYPE :** soil and sediment

**PRINCIPLE OF THE METHOD :**

Samples are dried, ground and homogenized. A known quantity of isotopically labelled PCDDs and PCDFs is added to the sample to serve as an internal quantitaion standard. PCDDs and PCDFs are extracted from soil/sediment using a Soxhlet extraction apparatus and toluene. The concentrated extract is processed through a multi-stage chromatographic cleanup procedure to remove potential chemical interferences.

The reconstituted final extract is analyzed by gas chromatography - tandem mass spectrometry (GC-MS-MS) or gas chromatography - high resolution mass spectrometry (GC-HRMS).

Further cleanup using high performance liquid chromatography (HPLC) may be necessary prior to final analysis if the sample is highly contaminated with chemical interferences that are not removed by the open-column chromatographic cleanup.

**PARAMETERS MEASURED :**

**IDL ( pg/g )**

**MDL ( pg/g )**

|   |   |    |
|---|---|----|
| 2,3,7,8-tetrachlorodibenzo-p-dioxin       | 1 | 2  |
| 1,2,3,7,8-pentachlorodibenzo-p-dioxin     | 2 | 4  |
| 1,2,3,4,7,8-hexachlorodibenzo-p-dioxin    | 3 | 3  |
| 1,2,3,6,7,8-hexachlorodibenzo-p-dioxin    | 3 | 3  |
| 1,2,3,7,8,9-hexachlorodibenzo-p-dioxin    | 3 | 3  |
| 1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin | 5 | 5  |
| octachlorodibenzo-p-dioxin                | 7 | 7  |
| 2,3,7,8-tetrachlorodibenzofuran           | 1 | 2  |
| 2,3,4,7,8-pentachlorodibenzofuran         | 2 | 5  |
| 1,2,3,7,8-pentachlorodibenzofuran         | 2 | 4  |
| 1,2,3,4,7,8-hexachlorodibenzofuran        | 3 | 3  |
| 1,2,3,6,7,8-hexachlorodibenzofuran        | 3 | 4  |
| 2,3,4,6,7,8-hexachlorodibenzofuran        | 3 | 3  |
| 1,2,3,7,8,9-hexachlorodibenzofuran        | 3 | 3  |
| 1,2,3,4,6,7,8-heptachlorodibenzofuran     | 5 | 6  |
| 1,2,3,4,7,8,9-heptachlorodibenzofuran     | 5 | 8  |
| octachlorodibenzofuran                    | 7 | 10 |

( Parameters Measured continued )

total tetrachlorinated dibenzo-p-dioxins ( TCDD )  
total pentachlorinated dibenzo-p-dioxins ( PCDD )  
total hexachlorinated dibenzo-p-dioxins ( HxCDD )  
total heptachlorinated dibenzo-p-dioxins ( HpCDD )  
total tetrachlorinated dibenzofurans ( TCDF )  
total pentachlorinated dibenzofurans ( PCDF )  
total hexachlorinated dibenzofurans ( HxCDF )  
total heptachlorinated dibenzofurans ( HpCDF )

**REPORTING FORMAT :**

Results are reported as ppt ( picograms of CDD/CDF per gram of dry soil ) rounded off to 2 significant figures. The minimum reported levels are sample and analyte specific \* and range from 1 pg/g to 10 pg/g.

**QUALITY CONTROL :**

The routine quality control operations monitor overall method performance ( precision and recovery samples ), validity of calibration and consistency in injection volume ( injection standard ), absence of potential contamination ( method blanks ) and recovery of target analytes ( internal quantitation standard ).

**REMARKS :** The performance limits ( established at recoveries of 70% and 130% ) were adopted by the Dioxin Unit as the method performance control limits.

List of Performance Charts and Tables:

Method Blanks Summary

2,3,7,8-tetrachlorodibenzo-p-dioxin  
1,2,3,7,8-pentachlorodibenzo-p-dioxin  
1,2,3,4,7,8-hexachlorodibenzo-p-dioxin  
1,2,3,6,7,8-hexachlorodibenzo-p-dioxin  
1,2,3,7,8,9-hexachlorodibenzo-p-dioxin  
1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin  
octachlorodibenzo-p-dioxin

2,3,7,8-tetrachlorodibenzofuran  
2,3,4,7,8-pentachlorodibenzofuran  
1,2,3,7,8-pentachlorodibenzofuran  
1,2,3,4,7,8-hexachlorodibenzofuran

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\* The minimum reported levels correspond to the amount of analyte that would give most-abundant ion response five times higher than corresponding instrumental noise.

( List of Performance Charts and Tables continued )

1,2,3,6,7,8-hexachlorodibenzofuran  
2,3,4,6,7,8-hexachlorodibenzofuran  
1,2,3,7,8,9-hexachlorodibenzofuran  
1,2,3,4,6,7,8-heptachlorodibenzofuran  
1,2,3,4,7,8,9-heptachlorodibenzofuran  
octachlorodibenzofuran

Method Blanks Summary

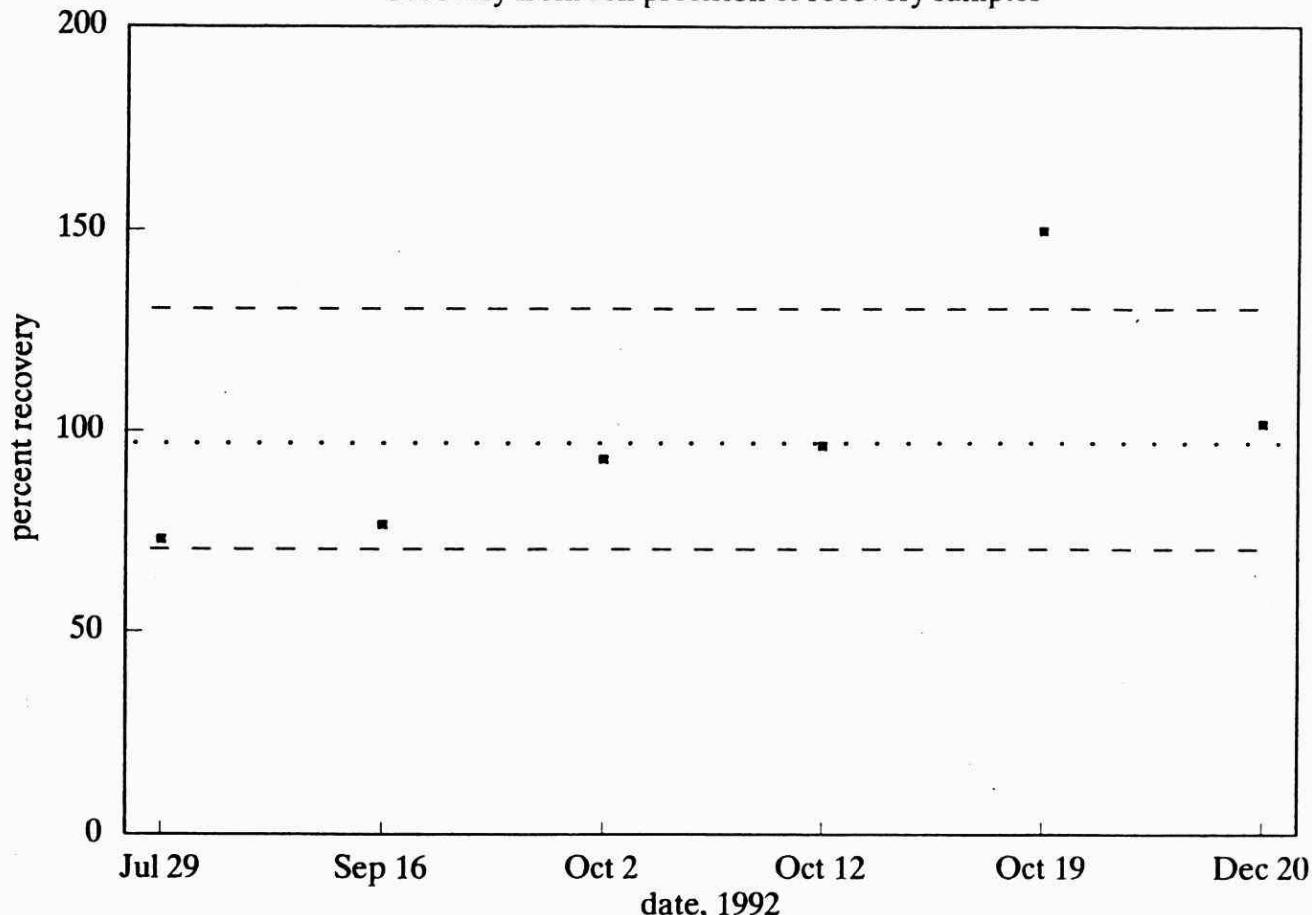
January 1992 - December 1992

| Analyte                                   | Number of Observations | Average Concentration ( pg/g ) | Standard Deviation ( pg/g ) |
|---|------------------------|--------------------------------|-----------------------------|
| 2,3,7,8-tetrachlorodibenzo-p-dioxin       | 14                     | 1.1                            | 3.2                         |
| 1,2,3,7,8-pentachlorodibenzo-p-dioxin     | 14                     | 0.07                           | 0.26                        |
| 1,2,3,4,7,8-hexachlorodibenzo-p-dioxin    | 14                     | ND ( 3 )                       |                             |
| 1,2,3,6,7,8-hexachlorodibenzo-p-dioxin    | 14                     | ND ( 3 )                       |                             |
| 1,2,3,7,8,9-hexachlorodibenzo-p-dioxin    | 14                     | ND ( 3 )                       |                             |
| 1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin | 14                     | 1.2                            | 3.8                         |
| octachlorodibenzo-p-dioxin                | 14                     | 3.3                            | 9.1                         |
| 2,3,7,8-tetrachlorodibenzofuran           | 14                     | 0.12                           | 0.44                        |
| 2,3,4,7,8-pentachlorodibenzofuran         | 14                     | ND ( 2 )                       |                             |
| 1,2,3,7,8-pentachlorodibenzofuran         | 14                     | ND ( 2 )                       |                             |
| 1,2,3,4,7,8-hexachlorodibenzofuran        | 14                     | 2.3                            | 8.3                         |
| 1,2,3,6,7,8-hexachlorodibenzofuran        | 14                     | ND ( 3 )                       |                             |
| 2,3,4,6,7,8-hexachlorodibenzofuran        | 14                     | ND ( 3 )                       |                             |
| 1,2,3,7,8,9-hexachlorodibenzofuran        | 14                     | ND ( 3 )                       |                             |
| 1,2,3,4,6,7,8-heptachlorodibenzofuran     | 14                     | 5                              | 13                          |
| 1,2,3,4,7,8,9-heptachlorodibenzofuran     | 14                     | ND ( 5 )                       |                             |
| octachlorodibenzofuran                    | 14                     | 10                             | 24                          |

ND ... Not detected. Detection limits pg/g given in brackets ( ).

## 2,3,7,8-tetrachlorodibenzo-p-dioxin

recovery from soil precision & recovery samples



..... average recovery ( % of expected )  
- - - - - control limits

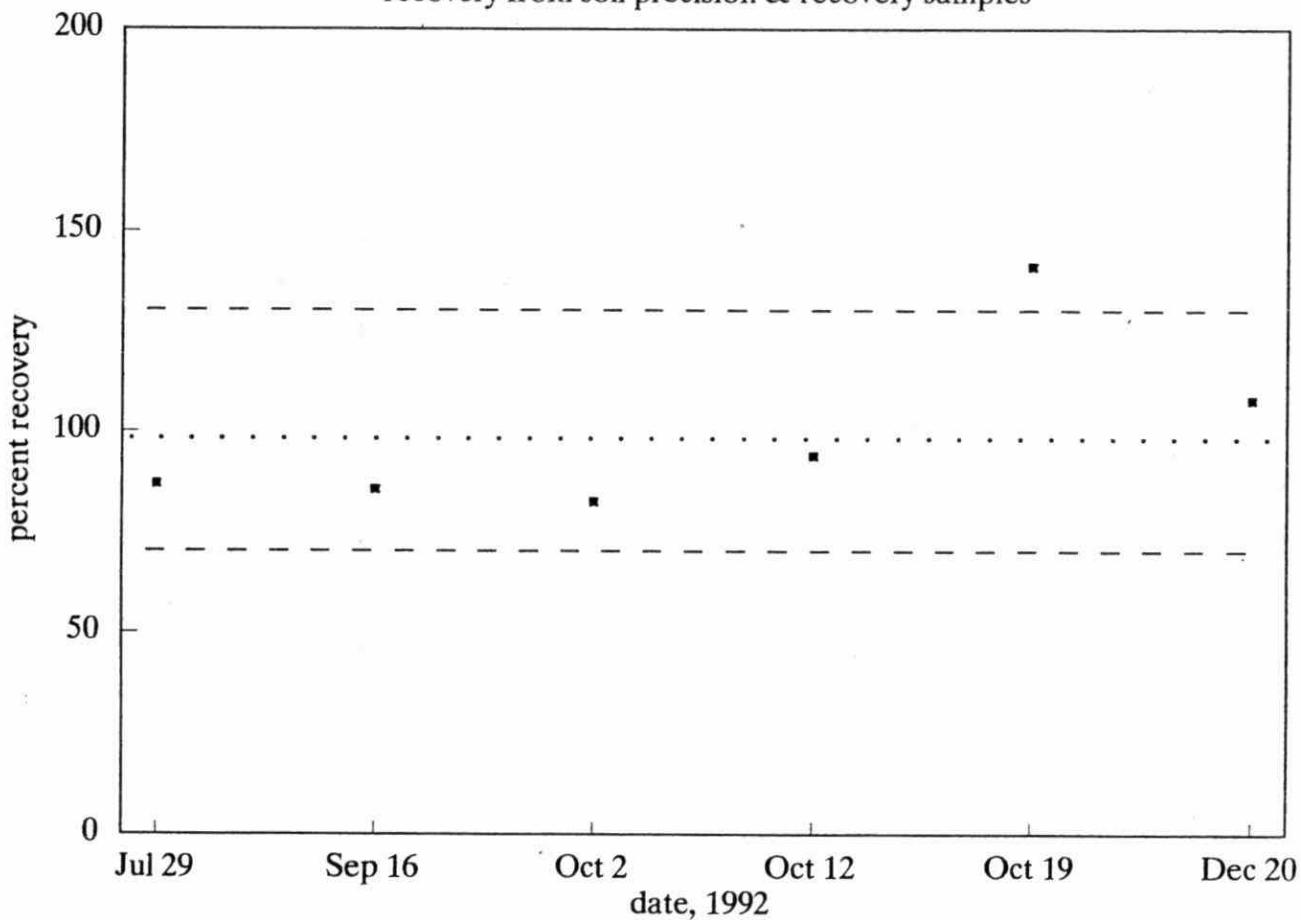
Performance Summary Table

January - December 1992

|                                |                                     |
|--------------------------------|-------------------------------------|
| Analyte                        | 2,3,7,8-tetrachlorodibenzo-p-dioxin |
| True Concentration             | 460 pg/g                            |
| Number of Observations         | 6                                   |
| Between-run Standard Deviation | 28 %                                |
| Accuracy (% of expected)       | 98 %                                |

## 1,2,3,7,8-pentachlorodibenzo-p-dioxin

recovery from soil precision & recovery samples



..... average recovery ( % of expected )  
- - - - - control limits

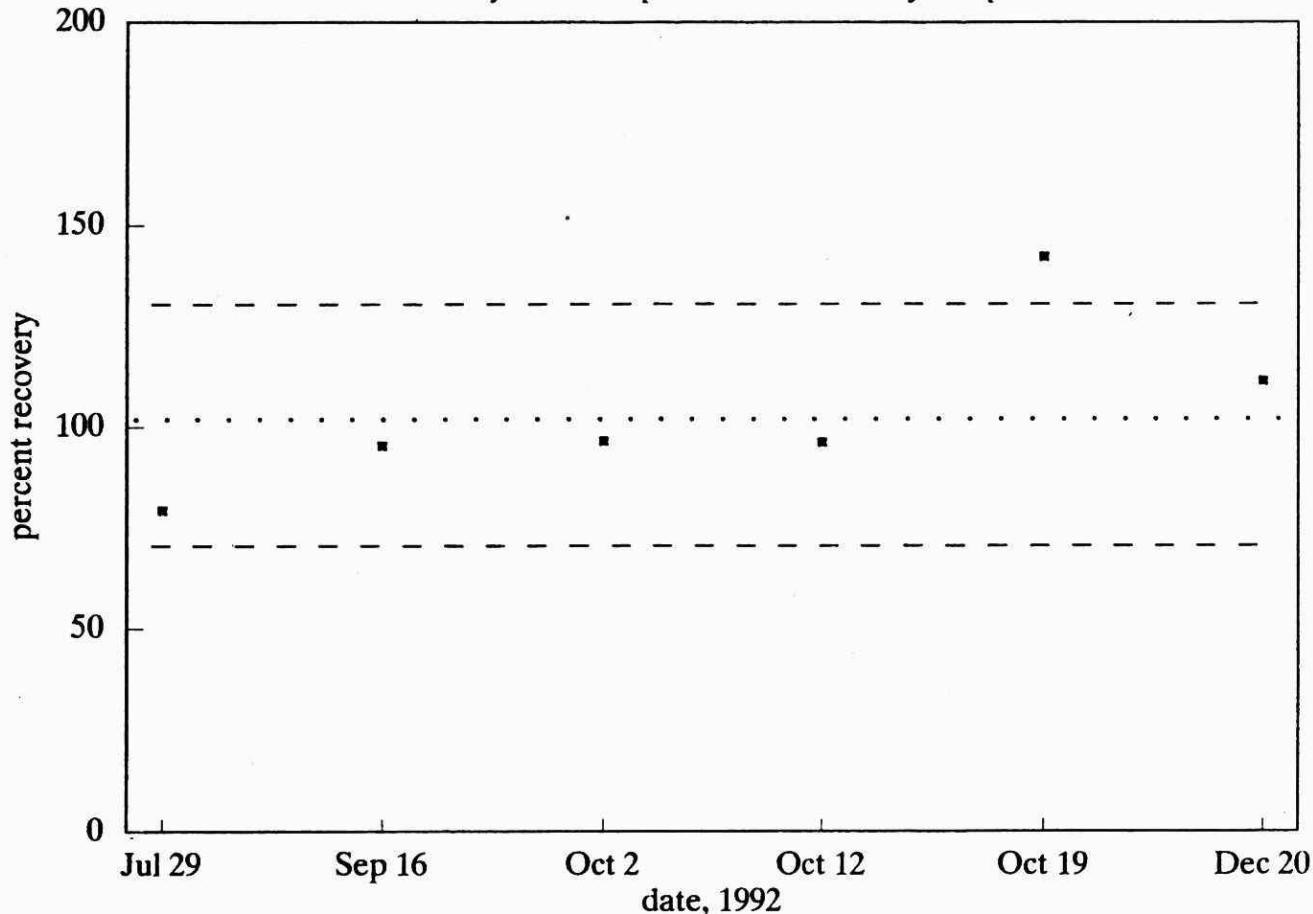
Performance Summary Table

January - December 1992

|                                |                                       |
|--------------------------------|---------------------------------------|
| Analyte                        | 1,2,3,7,8-pentachlorodibenzo-p-dioxin |
| True Concentration             | 960 pg/g                              |
| Number of Observations         | 6                                     |
| Between-run Standard Deviation | 22 %                                  |
| Accuracy (% of expected)       | 100 %                                 |

## 1,2,3,4,7,8-hexachlorodibenzo-p-dioxin

recovery from soil precision & recovery samples



..... average recovery ( % of expected )  
- - - - - control limits

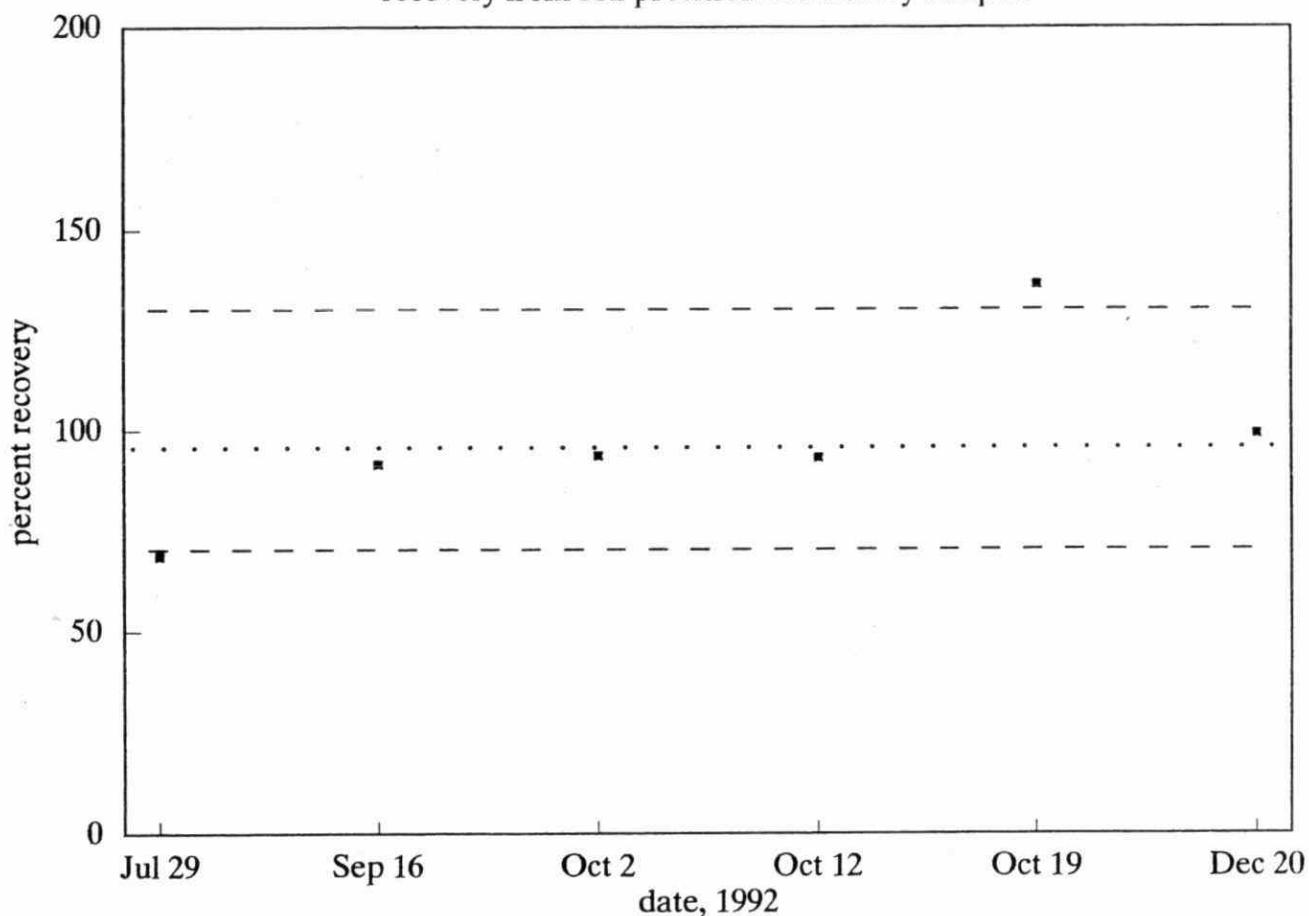
Performance Summary Table

January - December 1992

|                                |  |
|--------------------------------|--|
| Analyte                        | 1,2,3,4,7,8-hexachlorodibenzo-p-dioxin |
| True Concentration             | 900 pg/g                               |
| Number of Observations         | 6                                      |
| Between-run Standard Deviation | 22 %                                   |
| Accuracy (% of expected)       | 103 %                                  |

## 1,2,3,6,7,8-hexachlorodibenzo-p-dioxin

recovery from soil precision & recovery samples



..... average recovery ( % of expected )  
- - - - - control limits

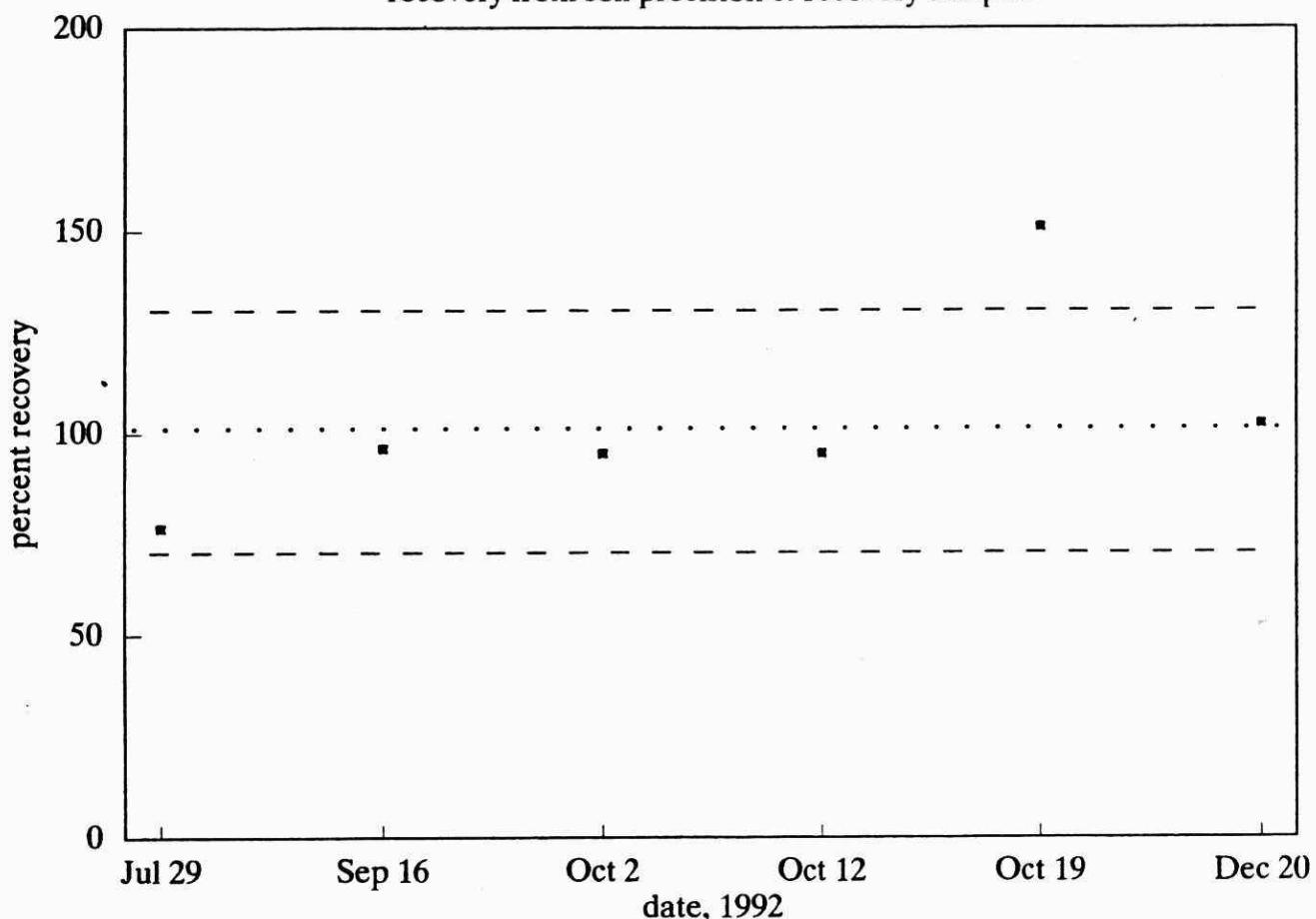
Performance Summary Table

January - December 1992

|                                |  |
|--------------------------------|--|
| Analyte                        | 1,2,3,6,7,8-hexachlorodibenzo-p-dioxin |
| True Concentration             | 870 pg/g                               |
| Number of Observations         | 6                                      |
| Between-run Standard Deviation | 22 %                                   |
| Accuracy (% of expected)       | 97 %                                   |

## 1,2,3,7,8,9-hexachlorodibenzo-p-dioxin

recovery from soil precision & recovery samples



..... average recovery ( % of expected )  
- - - - - control limits

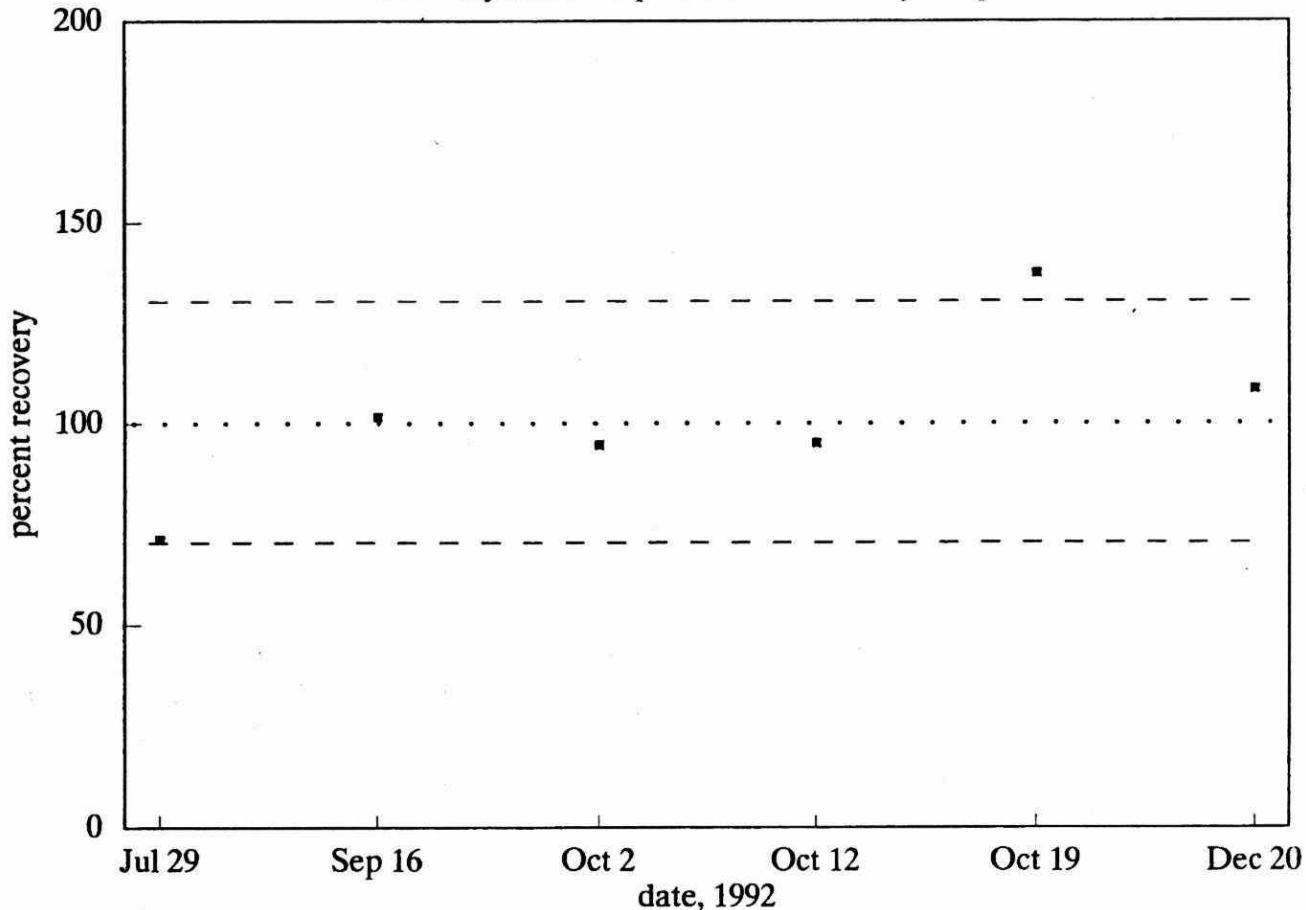
Performance Summary Table

January - December 1992

|                                |  |
|--------------------------------|--|
| Analyte                        | 1,2,3,7,8,9-hexachlorodibenzo-p-dioxin |
| True Concentration             | 900 pg/g                               |
| Number of Observations         | 6                                      |
| Between-run Standard Deviation | 25 %                                   |
| Accuracy (% of expected)       | 103 %                                  |

## 1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin

recovery from soil precision & recovery samples



..... average recovery ( % of expected )  
----- control limits

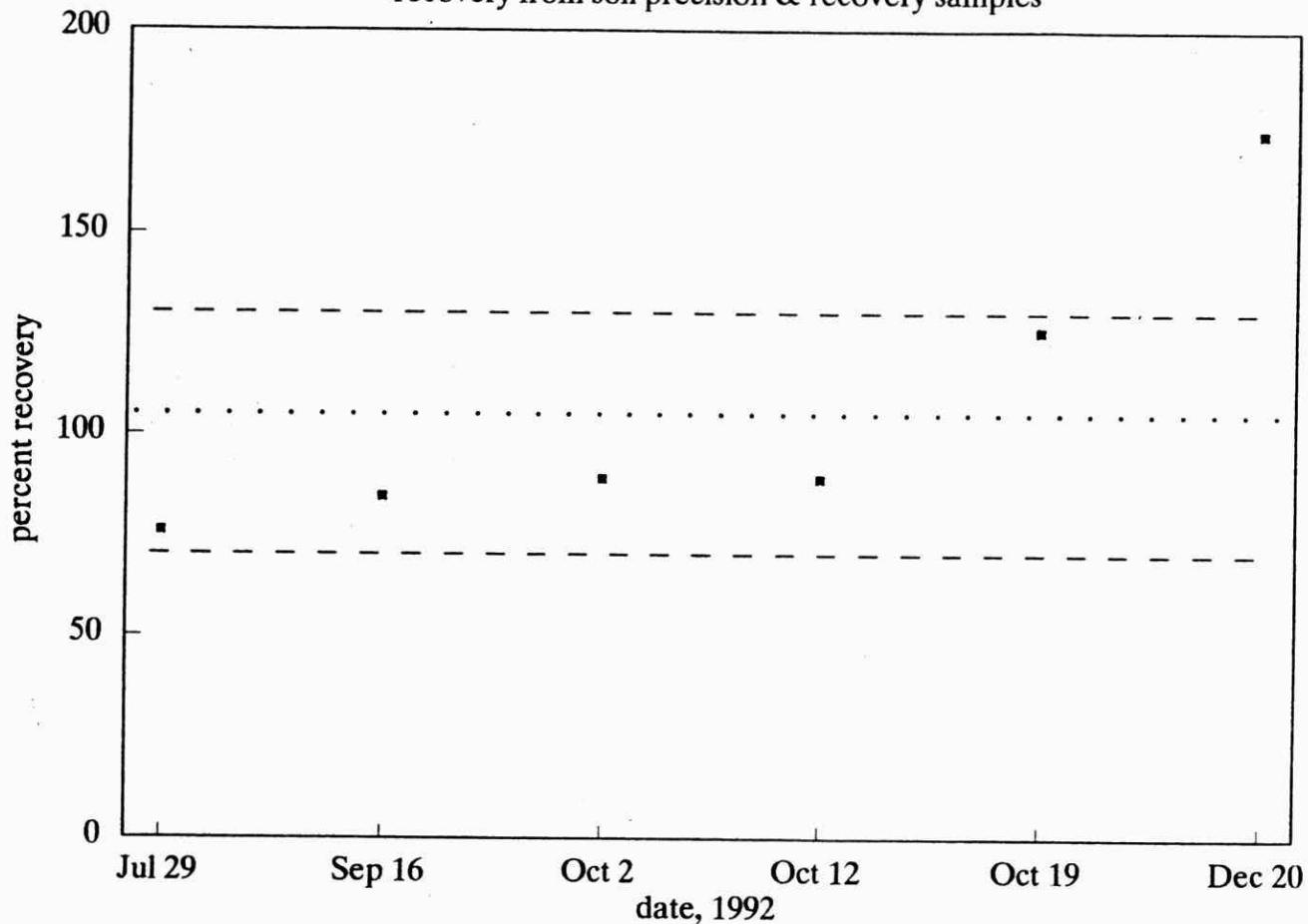
Performance Summary Table

January - December 1992

|                                |   |
|--------------------------------|---|
| Analyte                        | 1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin |
| True Concentration             | 1390 pg/g                                 |
| Number of Observations         | 6   |
| Between-run Standard Deviation | 22 %                                      |
| Accuracy (% of expected)       | 101 %                                     |

## octachlorodibenzo-p-dioxin

recovery from soil precision & recovery samples



..... average recovery ( % of expected )  
- - - - - control limits

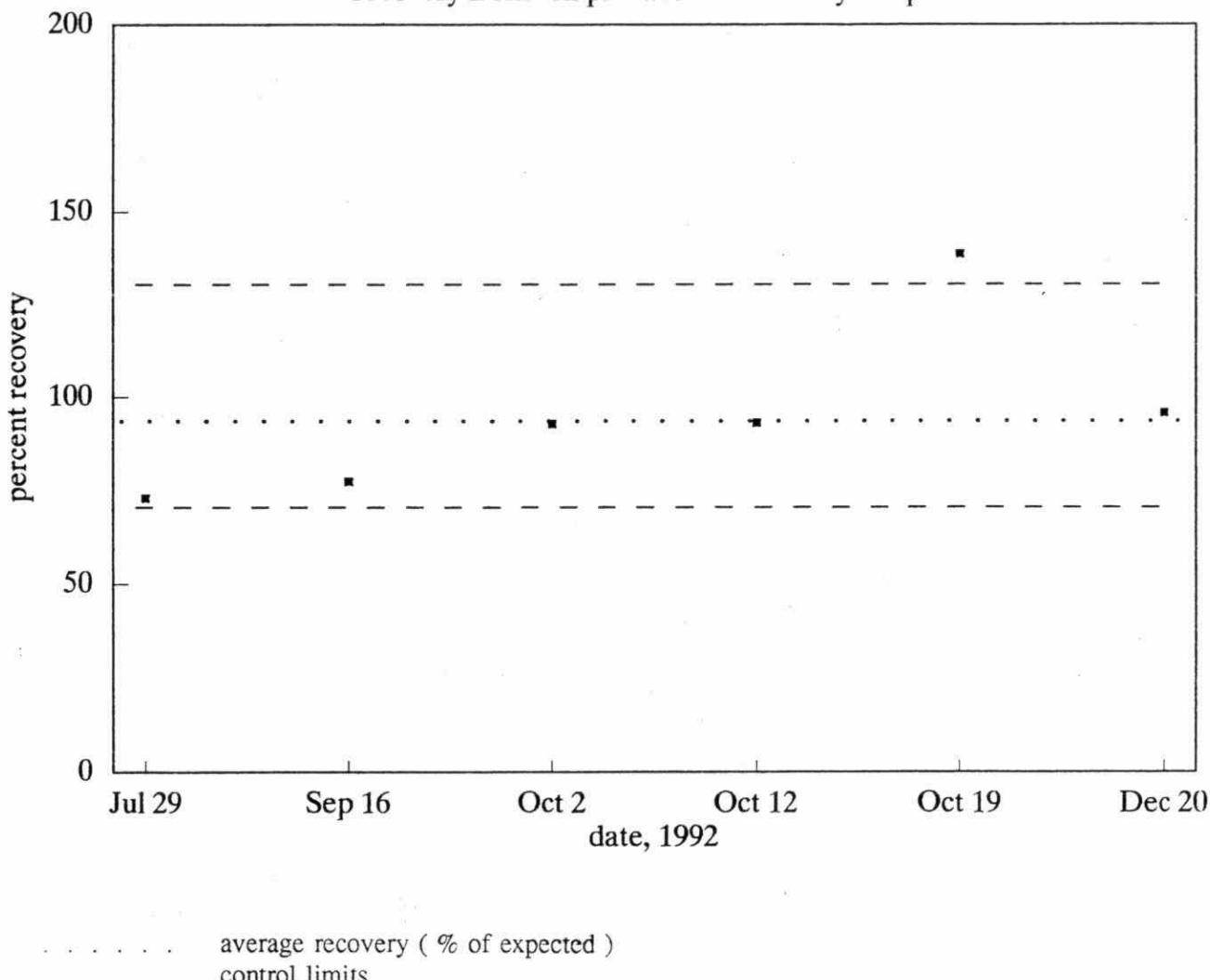
Performance Summary Table

January - December 1992

|                                |                            |
|--------------------------------|----------------------------|
| Analyte                        | octachlorodibenzo-p-dioxin |
| True Concentration             | 3510 pg/g                  |
| Number of Observations         | 6                          |
| Between-run Standard Deviation | 37 %                       |
| Accuracy (% of expected)       | 106 %                      |

## 2,3,7,8-tetrachlorodibenzofuran

recovery from soil precision & recovery samples



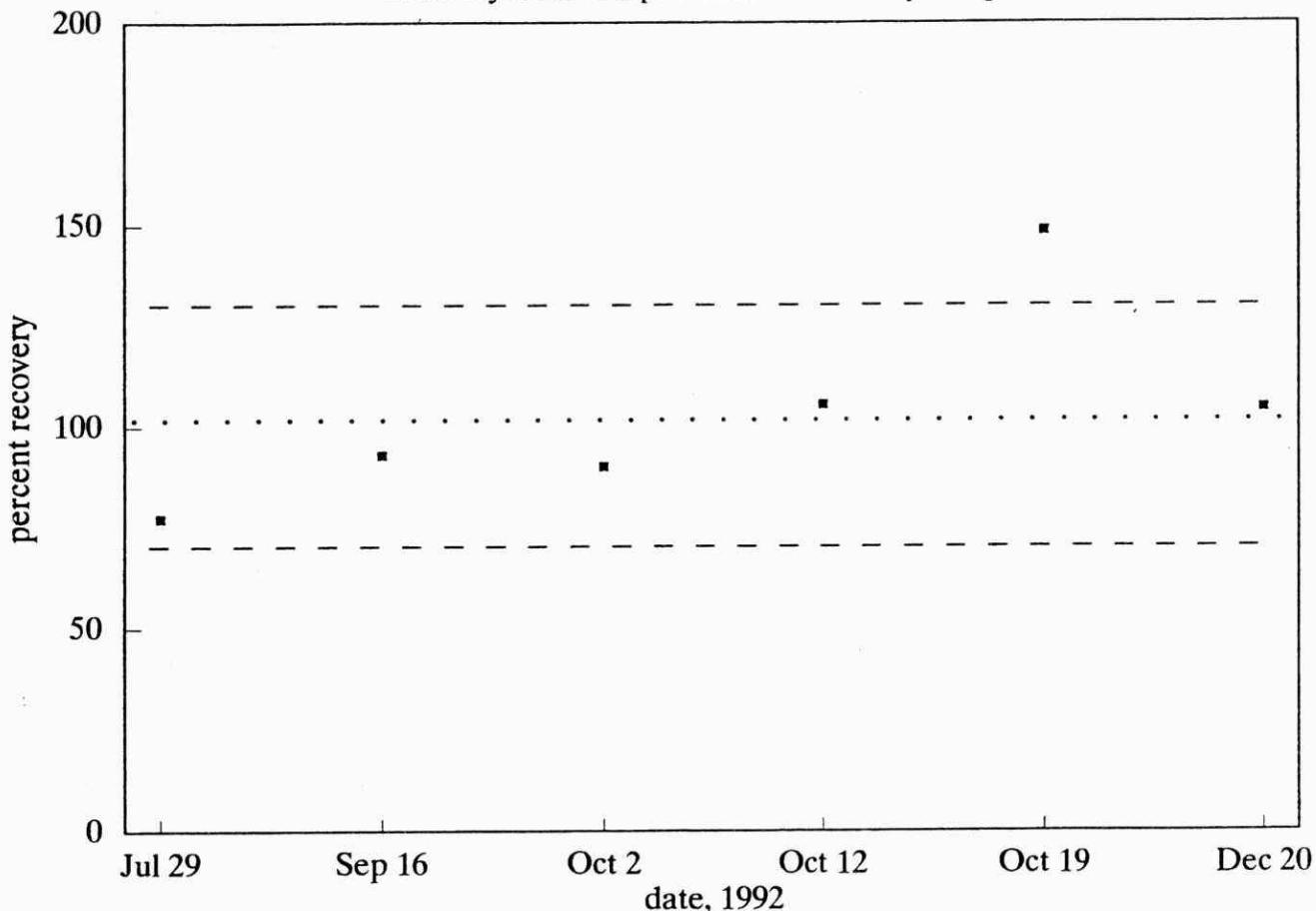
Performance Summary Table

January - December 1992

|                                |                                 |
|--------------------------------|---------------------------------|
| Analyte                        | 2,3,7,8-tetrachlorodibenzofuran |
| True Concentration             | 450 pg/g                        |
| Number of Observations         | 6                               |
| Between-run Standard Deviation | 23 %                            |
| Accuracy (% of expected)       | 95 %                            |

## 2,3,4,7,8-pentachlorodibenzofuran

recovery from soil precision & recovery samples



..... average recovery ( % of expected )  
- - - - - control limits

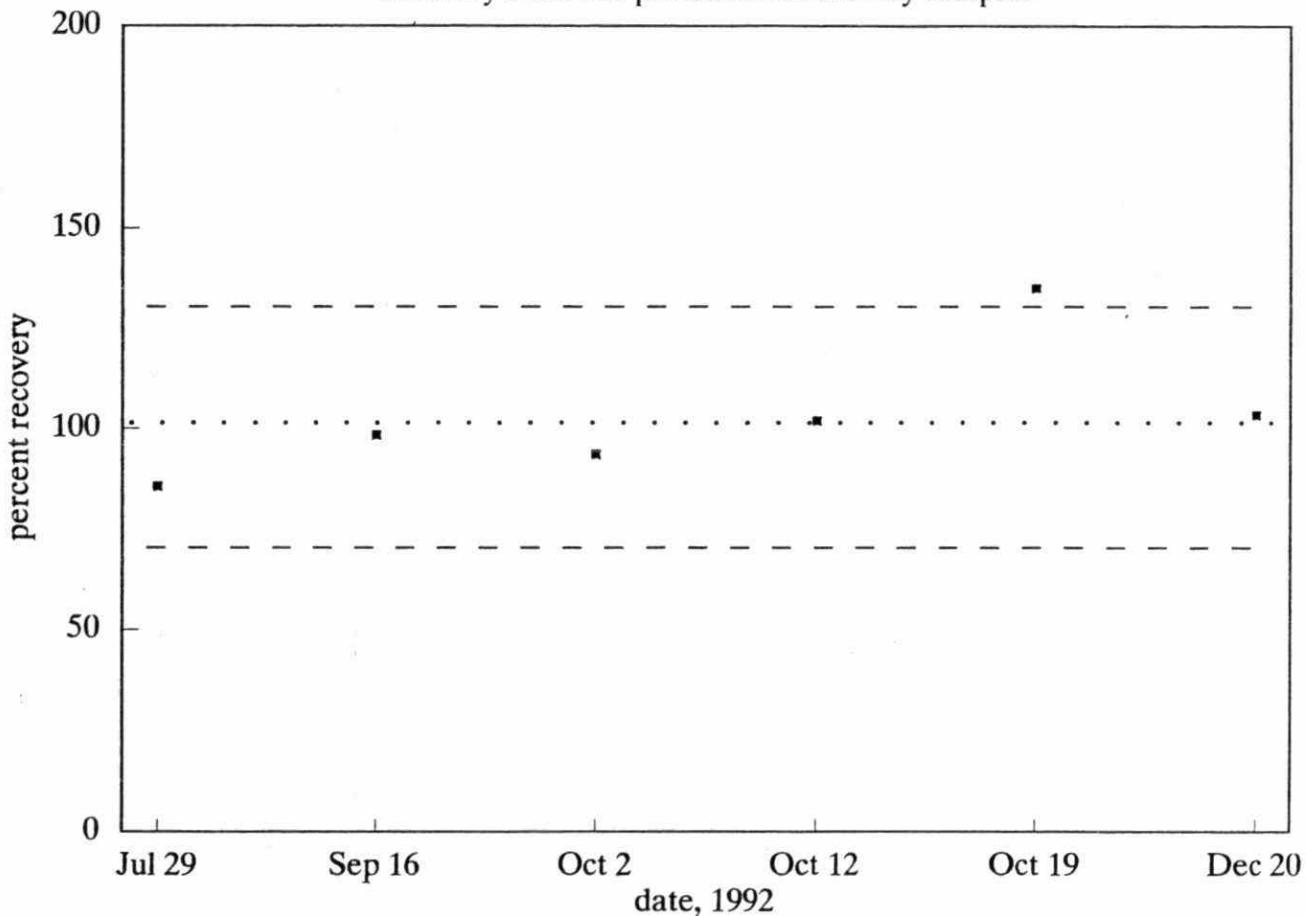
Performance Summary Table

January - December 1992

|                                |                                   |
|--------------------------------|-----------------------------------|
| Analyte                        | 2,3,4,7,8-pentachlorodibenzofuran |
| True Concentration             | 870 pg/g                          |
| Number of Observations         | 6                                 |
| Between-run Standard Deviation | 25 %                              |
| Accuracy (% of expected)       | 103 %                             |

## 1,2,3,7,8-pentachlorodibenzofuran

recovery from soil precision & recovery samples



..... average recovery ( % of expected )  
- - - - - control limits

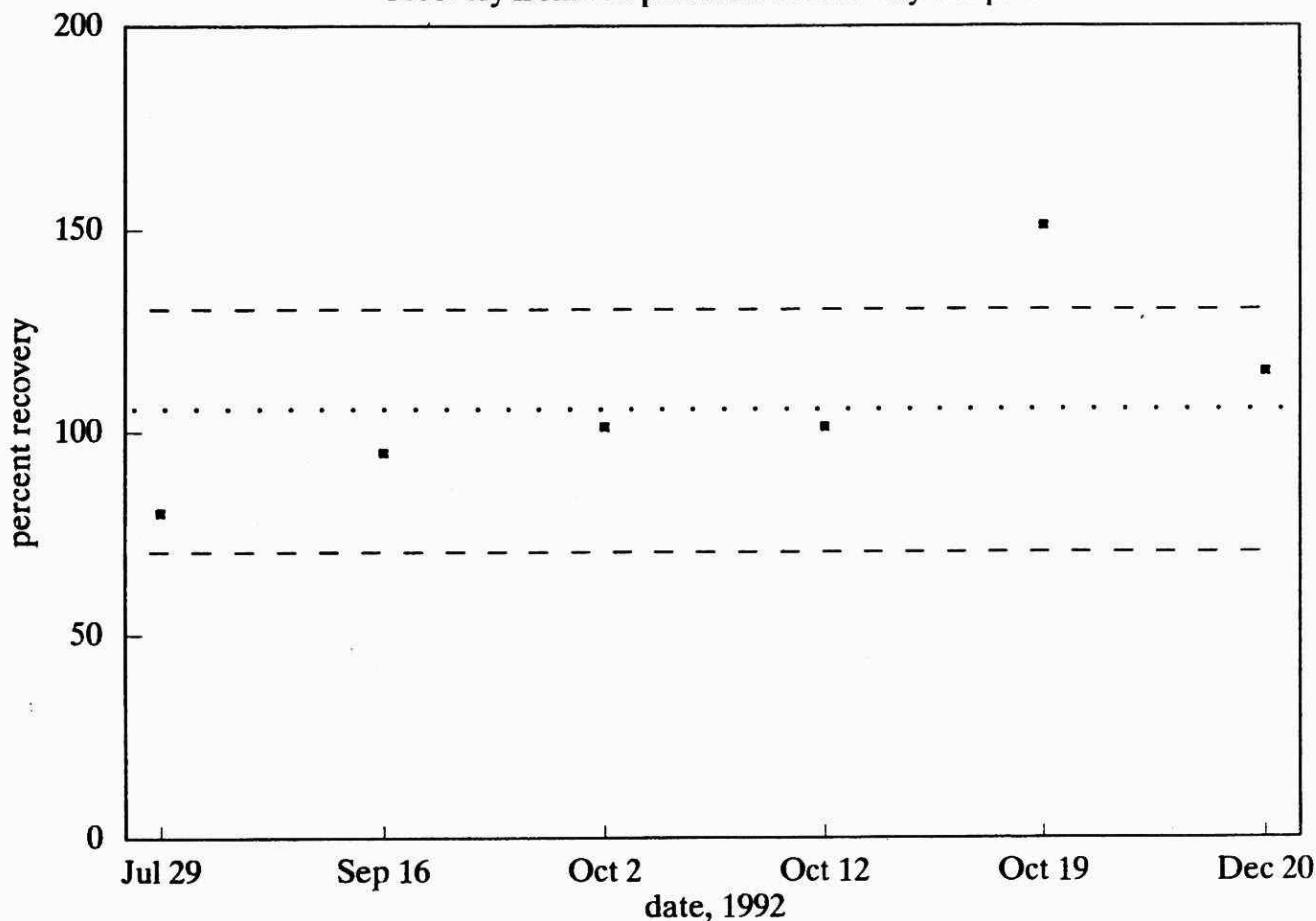
Performance Summary Table

January - December 1992

|                                |                                   |
|--------------------------------|-----------------------------------|
| Analyte                        | 1,2,3,7,8-pentachlorodibenzofuran |
| True Concentration             | 860 pg/g                          |
| Number of Observations         | 6                                 |
| Between-run Standard Deviation | 17 %                              |
| Accuracy (% of expected)       | 103 %                             |

## 1,2,3,4,7,8-hexachlorodibenzofuran

recovery from soil precision & recovery samples



..... average recovery ( % of expected )  
- - - - - control limits

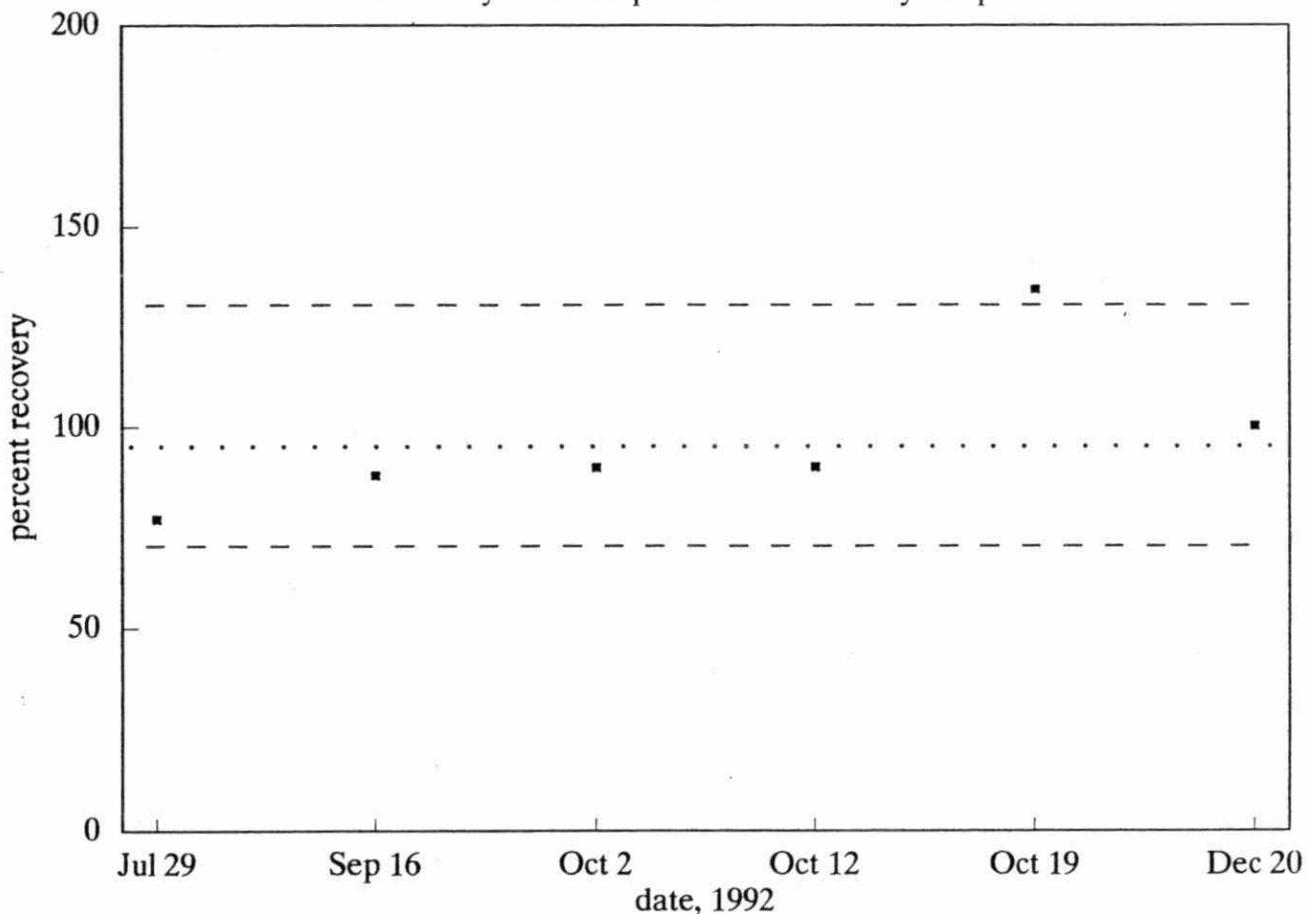
Performance Summary Table

January - December 1992

|                                |                                    |
|--------------------------------|------------------------------------|
| Analyte                        | 1,2,3,4,7,8-hexachlorodibenzofuran |
| True Concentration             | 880 pg/g                           |
| Number of Observations         | 6                                  |
| Between-run Standard Deviation | 24 %                               |
| Accuracy (% of expected)       | 107 %                              |

## 1,2,3,6,7,8-hexachlorodibenzofuran

recovery from soil precision & recovery samples



..... average recovery ( % of expected )  
- - - - - control limits

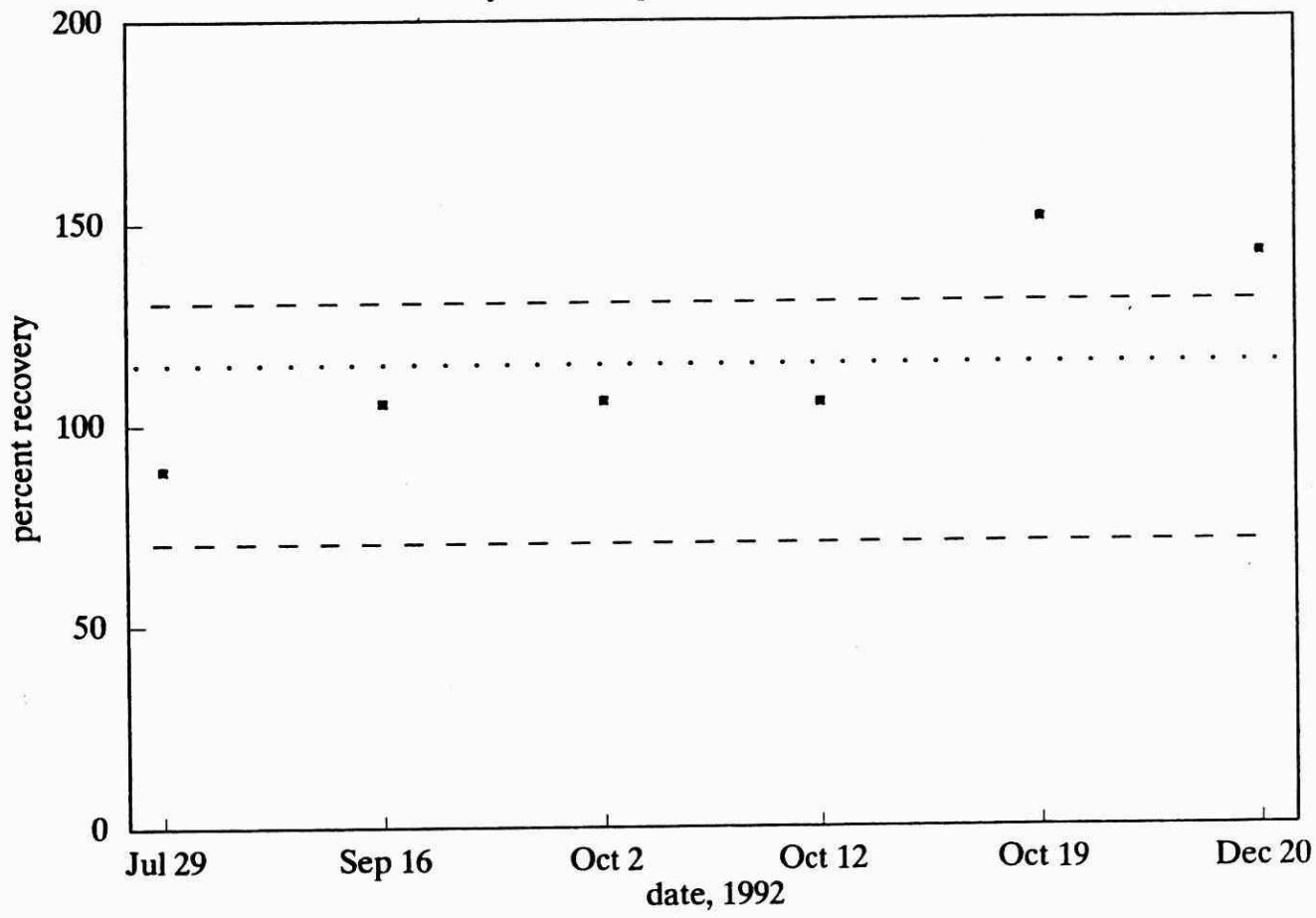
Performance Summary Table

January - December 1992

|                                |                                    |
|--------------------------------|------------------------------------|
| Analyte                        | 1,2,3,6,7,8-hexachlorodibenzofuran |
| True Concentration             | 950 pg/g                           |
| Number of Observations         | 6                                  |
| Between-run Standard Deviation | 20 %                               |
| Accuracy (% of expected)       | 97 %                               |

## 2,3,4,6,7,8-hexachlorodibenzofuran

recovery from soil precision & recovery samples



..... average recovery ( % of expected )  
- - - - - control limits

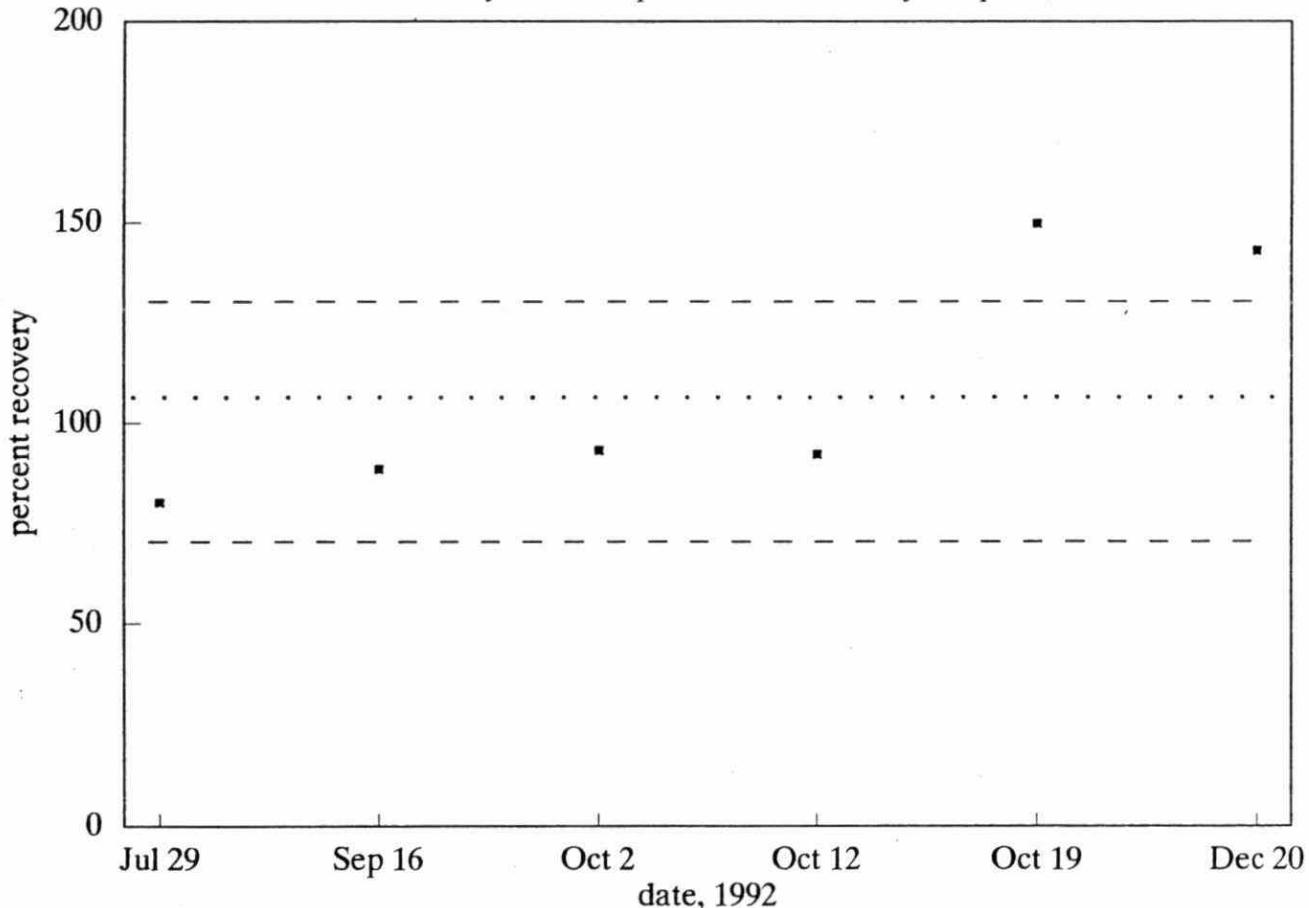
Performance Summary Table

January - December 1992

|                                |                                    |
|--------------------------------|------------------------------------|
| Analyte                        | 2,3,4,6,7,8-hexachlorodibenzofuran |
| True Concentration             | 820 pg/g                           |
| Number of Observations         | 6                                  |
| Between-run Standard Deviation | 24 %                               |
| Accuracy (% of expected)       | 116 %                              |

## 1,2,3,7,8,9-hexachlorodibenzofuran

recovery from soil precision & recovery samples



..... average recovery ( % of expected )  
- - - - - control limits

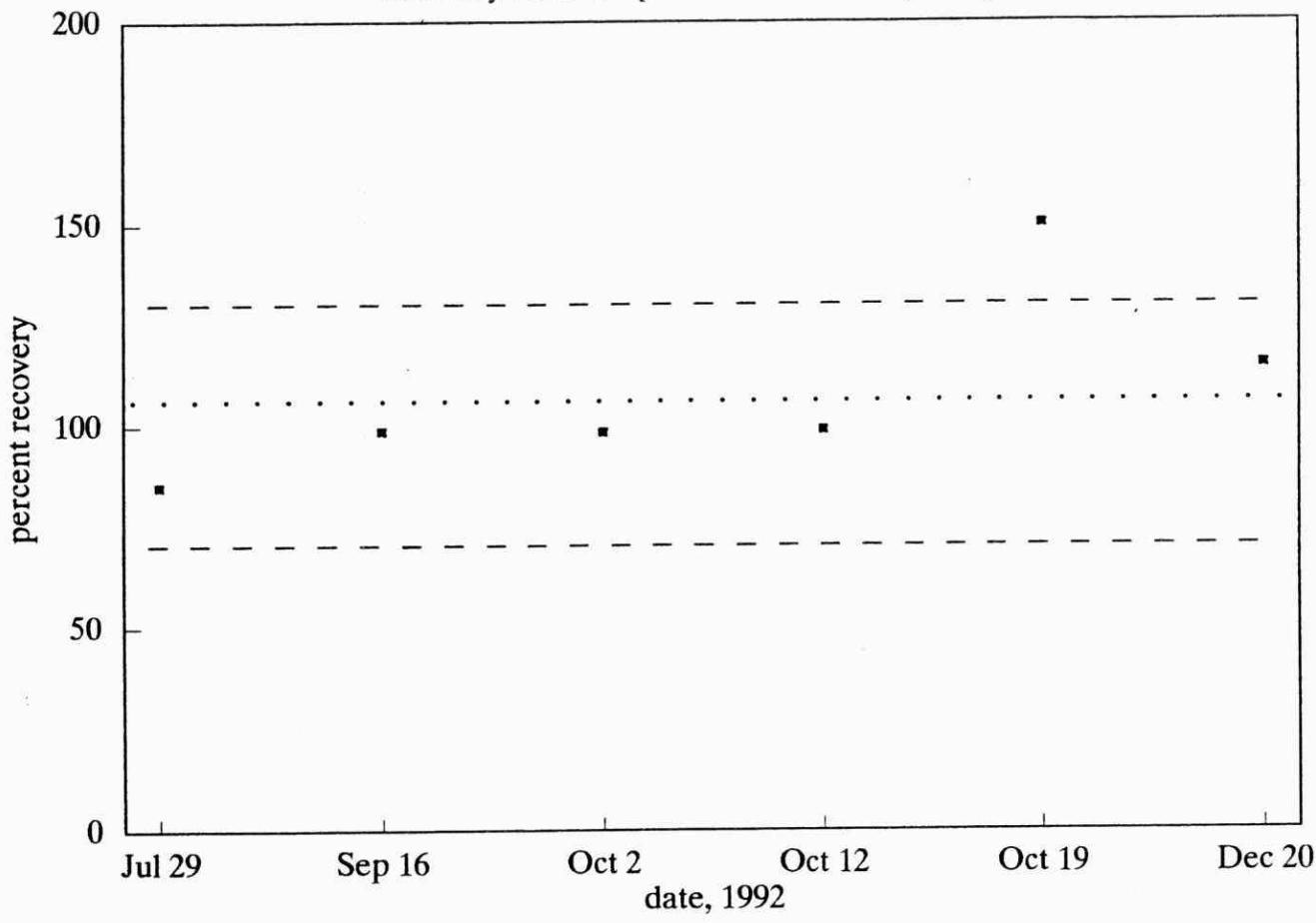
Performance Summary Table

January - December 1992

|                                |                                    |
|--------------------------------|------------------------------------|
| Analyte                        | 1,2,3,7,8,9-hexachlorodibenzofuran |
| True Concentration             | 910 pg/g                           |
| Number of Observations         | 6                                  |
| Between-run Standard Deviation | 30 %                               |
| Accuracy (% of expected)       | 108 %                              |

## 1,2,3,4,6,7,8-heptachlorodibenzofuran

recovery from soil precision & recovery samples



..... average recovery ( % of expected )  
- - - - - control limits

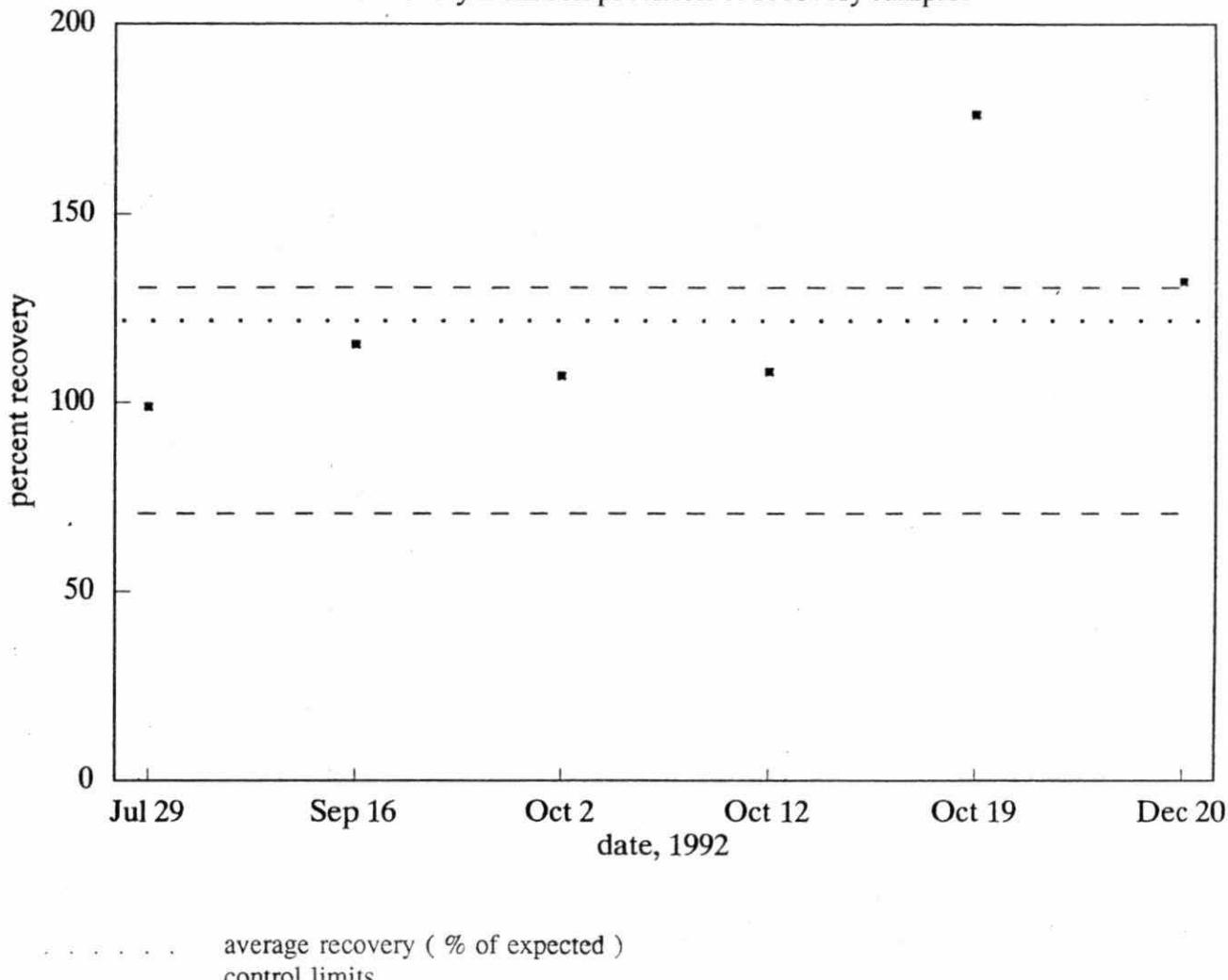
Performance Summary Table

January - December 1992

|                                |                                       |
|--------------------------------|---------------------------------------|
| Analyte                        | 1,2,3,4,6,7,8-heptachlorodibenzofuran |
| True Concentration             | 1270 pg/g                             |
| Number of Observations         | 6                                     |
| Between-run Standard Deviation | 23 %                                  |
| Accuracy (% of expected)       | 108 %                                 |

## 1,2,3,4,7,8,9-heptachlorodibenzofuran

recovery from soil precision & recovery samples



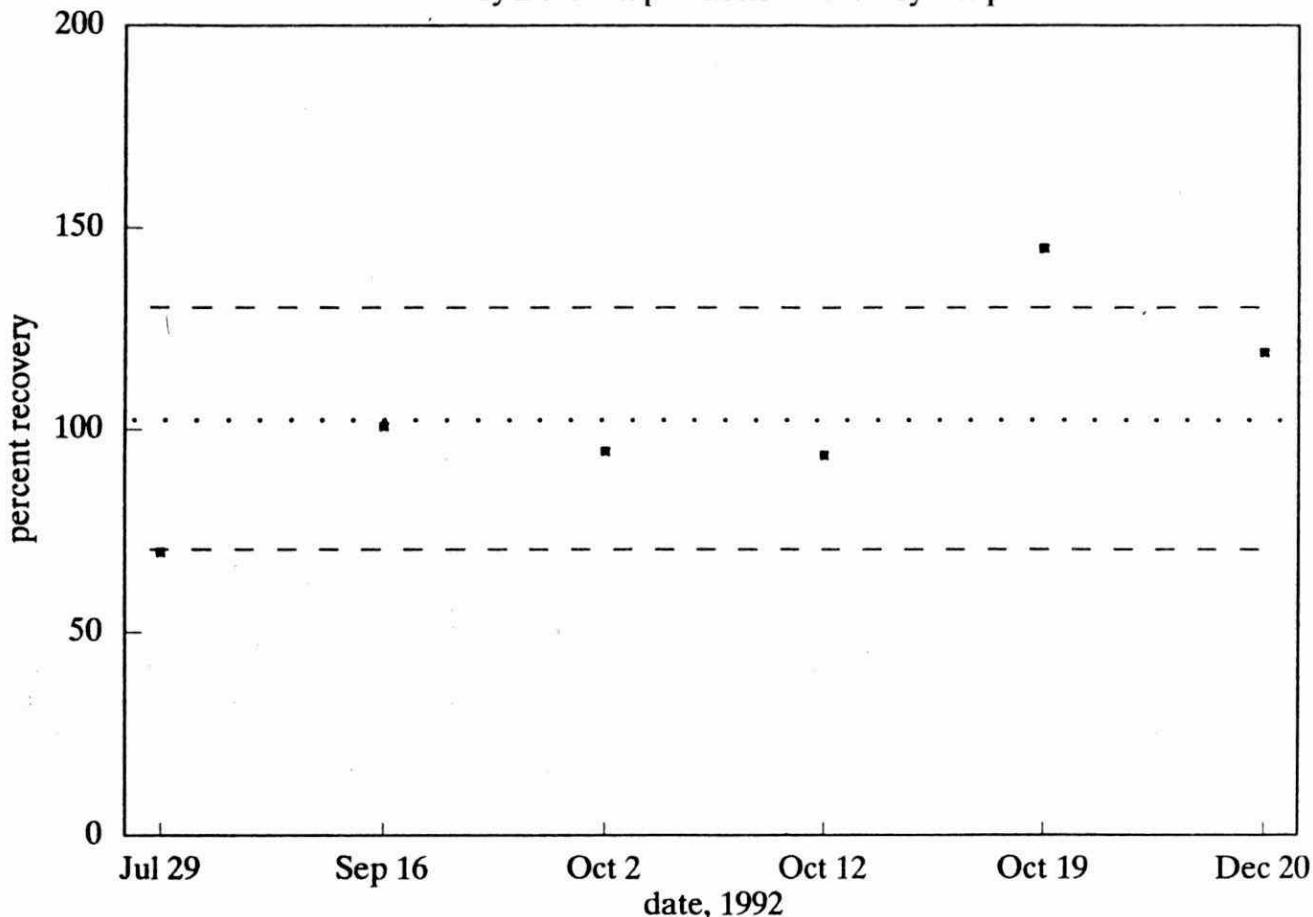
Performance Summary Table

January - December 1992

|                                |                                       |
|--------------------------------|---------------------------------------|
| Analyte                        | 1,2,3,4,7,8,9-heptachlorodibenzofuran |
| True Concentration             | 1120 pg/g                             |
| Number of Observations         | 6                                     |
| Between-run Standard Deviation | 28 %                                  |
| Accuracy (% of expected)       | 123 %                                 |

## octachlorodibenzofuran

recovery from soil precision & recovery samples



..... average recovery ( % of expected )  
- - - - - control limits

Performance Summary Table

January - December 1992

| Analyte                        | octachlorodibenzofuran |
|--------------------------------|------------------------|
| True Concentration             | 2250 pg/g              |
| Number of Observations         | 6                      |
| Between-run Standard Deviation | 26 %                   |
| Accuracy (% of expected)       | 104 %                  |

**METHOD CODE :** PWAFD-E3163B

**METHOD TITLE:** The Determination of Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans in Drinking Water by GC-MS

**LABORATORY :** Dioxin Unit

**SUPERVISOR :** Dr. E. Reiner

**SAMPLE TYPE :** raw or treated drinking water

**PRINCIPLE OF THE METHOD :**

A known quantity of isotopically labelled PCDDs and PCDFs is added to each sample to serve as an internal standard. Samples are filtered if they contain particulates. The particulate portion is solid/liquid extracted using a Soxhlet extractor, while the aqueous portion is liquid/liquid extracted. The extracts are re-combined before cleanup. A multi-stage chromatographic cleanup procedure is used to remove potential chemical interferences.

The reconstituted final extract is examined by gas chromatography - high resolution mass spectrometry (GC-HRMS) or gas chromatography/tandem mass spectrometry (GC-MS-MS).

**PARAMETERS MEASURED :** **IDL ( pg/L )**

|  |    |
|--|----|
| 2,3,7,8-tetrachlorodibenzo-p-dioxin                | 3  |
| 1,2,3,7,8-pentachlorodibenzo-p-dioxin              | 5  |
| 1,2,3,4,7,8-hexachlorodibenzo-p-dioxin             | 7  |
| 1,2,3,6,7,8-hexachlorodibenzo-p-dioxin             | 7  |
| 1,2,3,7,8,9-hexachlorodibenzo-p-dioxin             | 7  |
| 1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin          | 10 |
| octachlorodibenzo-p-dioxin                         | 10 |
| 2,3,7,8-tetrachlorodibenzofuran                    | 5  |
| 2,3,4,7,8-pentachlorodibenzofuran                  | 5  |
| 1,2,3,7,8-pentachlorodibenzofuran                  | 5  |
| 1,2,3,4,7,8-hexachlorodibenzofuran                 | 7  |
| 1,2,3,6,7,8-hexachlorodibenzofuran                 | 7  |
| 2,3,4,6,7,8-hexachlorodibenzofuran                 | 7  |
| 1,2,3,7,8,9-hexachlorodibenzofuran                 | 7  |
| 1,2,3,4,6,7,8-heptachlorodibenzofuran              | 10 |
| 1,2,3,4,7,8,9-heptachlorodibenzofuran              | 10 |
| octachlorodibenzofuran                             | 10 |
| total tetrachlorinated dibenzo-p-dioxins ( TCDD )  |    |
| total pentachlorinated dibenzo-p-dioxins ( PCDD )  |    |
| total hexachlorinated dibenzo-p-dioxins ( HxCDD )  |    |
| total heptachlorinated dibenzo-p-dioxins ( HpCDD ) |    |
| total tetrachlorinated dibenzofurans ( TCDF )      |    |
| total pentachlorinated dibenzofurans ( PCDF )      |    |
| total hexachlorinated dibenzofurans ( HxCDF )      |    |
| total heptachlorinated dibenzofurans ( HpCDF )     |    |

**REPORTING FORMAT :**

Results are reported in parts per quadrillion ( pg/L ) rounded off to 2 significant figures. The minimum reported levels are sample and analyte specific and range from 3 ppq to 10 ppq.

**QUALITY CONTROL :**

The routine quality control operations monitor overall method performance ( precision and recovery samples ), validity of calibration and consistency in injection volume ( injection standard ), absence of potential contamination ( method blanks ) and recovery of target analytes ( internal quantitation standard ).

**List of Performance Tables :** Method Blanks Summary

| Method Blanks Summary                     |                        | January 1992 - December 1992   |                             |  |
|---|------------------------|--------------------------------|-----------------------------|--|
| Analyte                                   | Number of Observations | Average Concentration ( pg/L ) | Standard Deviation ( pg/L ) |  |
| 2,3,7,8-tetrachlorodibenzo-p-dioxin       | 5                      | ND ( 3 )                       |                             |  |
| 1,2,3,7,8-pentachlorodibenzo-p-dioxin     | 5                      | ND ( 5 )                       |                             |  |
| 1,2,3,4,7,8-hexachlorodibenzo-p-dioxin    | 5                      | ND ( 7 )                       |                             |  |
| 1,2,3,6,7,8-hexachlorodibenzo-p-dioxin    | 5                      | ND ( 7 )                       |                             |  |
| 1,2,3,7,8,9-hexachlorodibenzo-p-dioxin    | 5                      | ND ( 7 )                       |                             |  |
| 1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin | 5                      | ND ( 10 )                      |                             |  |
| octachlorodibenzo-p-dioxin                | 5                      | 1.7                            | 3.3                         |  |
| 2,3,7,8-tetrachlorodibenzofuran           | 5                      | ND ( 5 )                       |                             |  |
| 2,3,4,7,8-pentachlorodibenzofuran         | 5                      | ND ( 5 )                       |                             |  |
| 1,2,3,7,8-pentachlorodibenzofuran         | 5                      | ND ( 5 )                       |                             |  |
| 1,2,3,4,7,8-hexachlorodibenzofuran        | 5                      | ND ( 7 )                       |                             |  |
| 1,2,3,6,7,8-hexachlorodibenzofuran        | 5                      | ND ( 7 )                       |                             |  |
| 2,3,4,6,7,8-hexachlorodibenzofuran        | 5                      | ND ( 7 )                       |                             |  |
| 1,2,3,7,8,9-hexachlorodibenzofuran        | 5                      | ND ( 7 )                       |                             |  |
| 1,2,3,4,6,7,8-heptachlorodibenzofuran     | 5                      | ND ( 10 )                      |                             |  |
| 1,2,3,4,7,8,9-heptachlorodibenzofuran     | 5                      | ND ( 10 )                      |                             |  |
| octachlorodibenzofuran                    | 5                      | 1.0                            | 2.0                         |  |

ND ... Not detected. Detection limit in pg/L given in brackets ( ).

\* The minimum reported levels correspond to the amount of analyte that would give most-abundant ion response five times higher than corresponding instrumental noise.

**METHOD CODE :** PWAFD-E3164B

**METHOD TITLE:** The Determination of Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans in Groundwater and Aqueous Effluent by GC-MS

**LABORATORY :** Dioxin Unit

**SUPERVISOR :** Dr. E. Reiner

**SAMPLE TYPE :** groundwater, aqueous industrial or municipal effluent

**PRINCIPLE OF THE METHOD :**

A known quantity of isotopically labelled PCDDs and PCDFs is added to each sample to serve as an internal quantitation standard. Sample is then filtered to separate the aqueous and particulate portions. The particulate portion is solid/liquid extracted using a Soxhlet extractor, while the aqueous portion is liquid/liquid extracted. The extracts are re-combined before cleanup. A multi-stage chromatographic cleanup procedure is used to remove potential chemical interferences.

The reconstituted final extract is examined by gas chromatography - high resolution mass spectrometry (GC-HRMS) or gas chromatography/tandem mass spectrometry (GC-MS-MS).

Further cleanup using high performance liquid chromatography (HPLC) may be necessary prior to final analysis if the sample is highly contaminated with chemical interferences that are not removed by the open-column chromatographic cleanup.

| <b>PARAMETERS MEASURED :</b>              | <b>IDL ( pg/L )</b> | <b>MDL ( pg/L )</b> |
|---|---------------------|---------------------|
| 2,3,7,8-tetrachlorodibenzo-p-dioxin       | 3                   | 3.9                 |
| 1,2,3,7,8-pentachlorodibenzo-p-dioxin     | 5                   | 15                  |
| 1,2,3,4,7,8-hexachlorodibenzo-p-dioxin    | 7                   | 11                  |
| 1,2,3,6,7,8-hexachlorodibenzo-p-dioxin    | 7                   | 15                  |
| 1,2,3,7,8,9-hexachlorodibenzo-p-dioxin    | 7                   | 28                  |
| 1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin | 10                  | 21                  |
| octachlorodibenzo-p-dioxin                | 10                  | 72                  |
| 2,3,7,8-tetrachlorodibenzofuran           | 5                   | 21                  |
| 2,3,4,7,8-pentachlorodibenzofuran         | 5                   | 19                  |
| 1,2,3,7,8-pentachlorodibenzofuran         | 5                   | 21                  |
| 1,2,3,4,7,8-hexachlorodibenzofuran        | 7                   | 15                  |
| 1,2,3,6,7,8-hexachlorodibenzofuran        | 7                   | 11                  |
| 2,3,4,6,7,8-hexachlorodibenzofuran        | 7                   | 22                  |
| 1,2,3,7,8,9-hexachlorodibenzofuran        | 7                   | 15                  |
| 1,2,3,4,6,7,8-heptachlorodibenzofuran     | 10                  | 28                  |
| 1,2,3,4,7,8,9-heptachlorodibenzofuran     | 10                  | 20                  |
| octachlorodibenzofuran                    | 10                  | 35                  |

( Parameters Measured continued )

total tetrachlorinated dibenzo-p-dioxins ( TCDD )  
total pentachlorinated dibenzo-p-dioxins ( PCDD )  
total hexachlorinated dibenzo-p-dioxins ( HxCDD )  
total heptachlorinated dibenzo-p-dioxins ( HpCDD )  
total tetrachlorinated dibenzofurans ( TCDF )  
total pentachlorinated dibenzofurans ( PCDF )  
total hexachlorinated dibenzofurans ( HxCDF )  
total heptachlorinated dibenzofurans ( HpCDF )

**REPORTING FORMAT :**

Results are reported in parts per quadrillion ( pg/L ) rounded off to 2 significant figures. The minimum reported levels are sample and analyte specific and range from 5 ppq to 10 ppq.

**QUALITY CONTROL :**

The routine quality control operations monitor overall method performance ( precision and recovery samples ), validity of calibration and consistency in injection volume ( injection standard ), absence of potential contamination ( method blanks ) and recovery of target analytes ( internal quantitation standard ).

**REMARKS :** Two types of performance limits are displayed on the performance charts. One set was statistically derived from the 1992 data; while the other ( established at recoveries of 70% and 130% ) was adopted by the Dioxin Unit as the method performance control limits.

List of Performance Charts and Tables:

Method Blanks Summary  
2,3,7,8-tetrachlorodibenzo-p-dioxin  
1,2,3,7,8-pentachlorodibenzo-p-dioxin  
1,2,3,4,7,8-hexachlorodibenzo-p-dioxin  
1,2,3,6,7,8-hexachlorodibenzo-p-dioxin  
1,2,3,7,8,9-hexachlorodibenzo-p-dioxin  
1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin  
octachlorodibenzo-p-dioxin  
2,3,7,8-tetrachlorodibenzofuran  
2,3,4,7,8-pentachlorodibenzofuran  
1,2,3,7,8-pentachlorodibenzofuran  
1,2,3,4,7,8-hexachlorodibenzofuran  
1,2,3,6,7,8-hexachlorodibenzofuran  
2,3,4,6,7,8-hexachlorodibenzofuran

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\* The minimum reported levels correspond to the amount of analyte that would give most-abundant ion response five times higher than corresponding instrumental noise.

( List of Performance Charts and Tables continued )

1,2,3,7,8,9-hexachlorodibenzofuran  
1,2,3,4,6,7,8-heptachlorodibenzofuran  
1,2,3,4,7,8,9-heptachlorodibenzofuran  
octachlorodibenzofuran

Method Blanks Summary

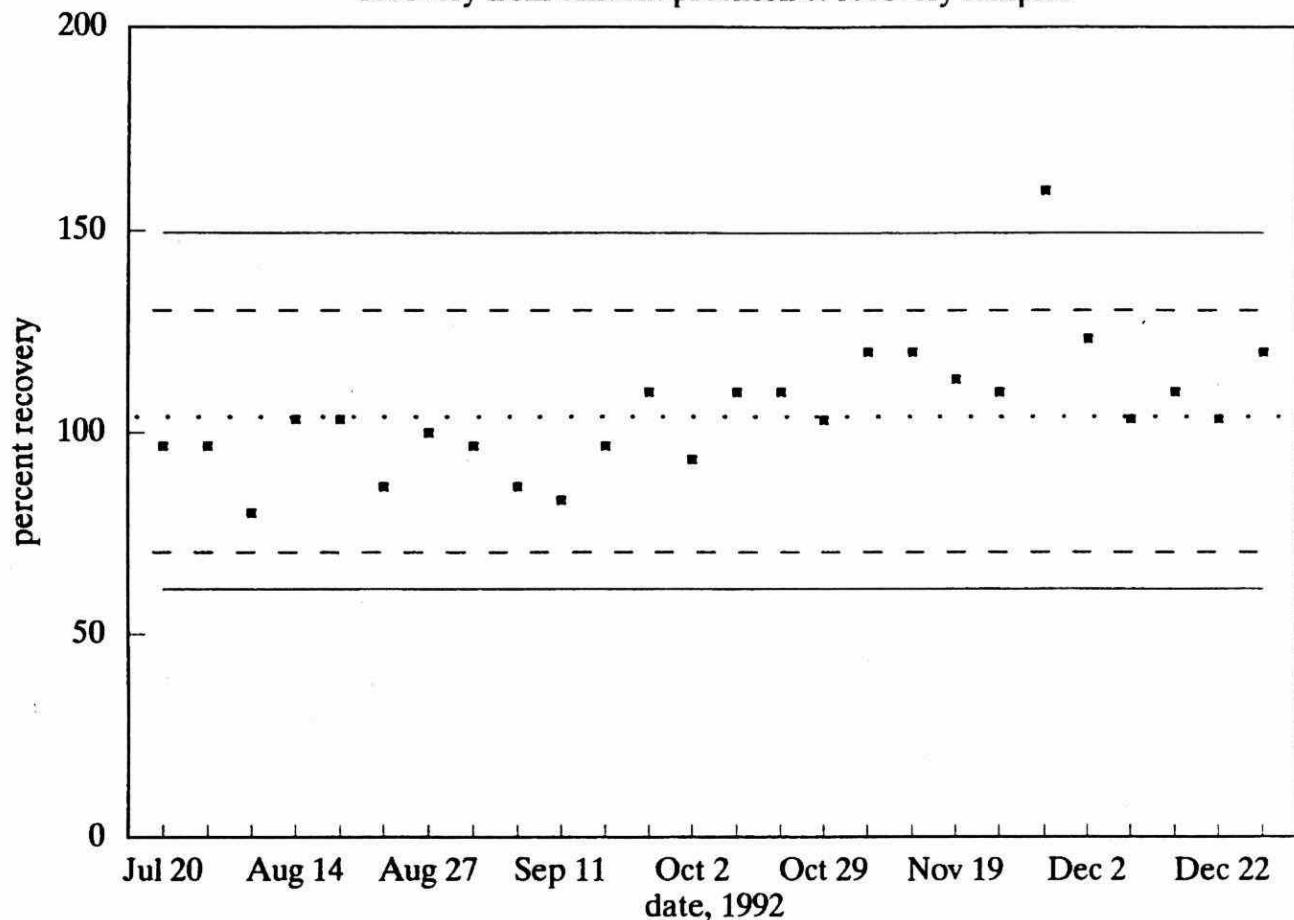
January 1992 - December 1992

| Analyte                                   | Number of Observations | Average Concentration ( pg/L ) | Standard Deviation ( pg/L ) |
|---|------------------------|--------------------------------|-----------------------------|
| 2,3,7,8-tetrachlorodibenzo-p-dioxin       | 38                     | 0.011                          | 0.06                        |
| 1,2,3,7,8-pentachlorodibenzo-p-dioxin     | 38                     | ND ( 5 )                       |                             |
| 1,2,3,4,7,8-hexachlorodibenzo-p-dioxin    | 38                     | ND ( 7 )                       |                             |
| 1,2,3,6,7,8-hexachlorodibenzo-p-dioxin    | 38                     | ND ( 7 )                       |                             |
| 1,2,3,7,8,9-hexachlorodibenzo-p-dioxin    | 38                     | ND ( 7 )                       |                             |
| 1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin | 38                     | 0.04                           | 0.26                        |
| octachlorodibenzo-p-dioxin                | 38                     | 1.1                            | 2.9                         |
| 2,3,7,8-tetrachlorodibenzofuran           | 38                     | ND ( 5 )                       |                             |
| 2,3,4,7,8-pentachlorodibenzofuran         | 38                     | ND ( 5 )                       |                             |
| 1,2,3,7,8-pentachlorodibenzofuran         | 38                     | ND ( 5 )                       |                             |
| 1,2,3,4,7,8-hexachlorodibenzofuran        | 38                     | ND ( 7 )                       |                             |
| 1,2,3,6,7,8-hexachlorodibenzofuran        | 38                     | ND ( 7 )                       |                             |
| 2,3,4,6,7,8-hexachlorodibenzofuran        | 38                     | 0.04                           | 0.25                        |
| 1,2,3,7,8,9-hexachlorodibenzofuran        | 38                     | ND ( 7 )                       |                             |
| 1,2,3,4,6,7,8-heptachlorodibenzofuran     | 38                     | ND ( 10 )                      |                             |
| 1,2,3,4,7,8,9-heptachlorodibenzofuran     | 38                     | ND ( 10 )                      |                             |
| octachlorodibenzofuran                    | 38                     | 0.13                           | 0.80                        |

ND ... Not detected. Detection limits in pg/L given in brackets ( ).

## 2,3,7,8-tetrachlorodibenzo-p-dioxin

recovery from effluent precision & recovery samples



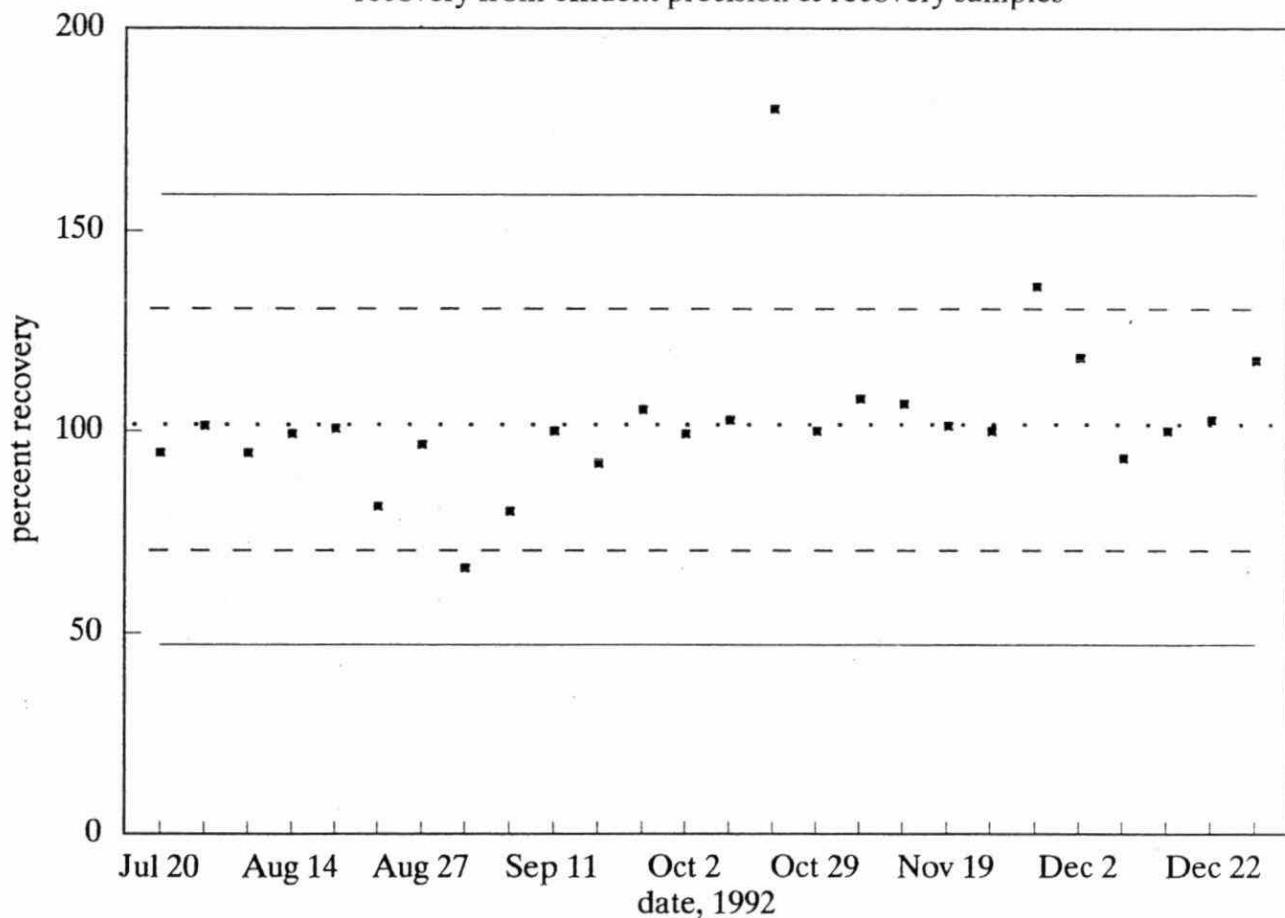
Performance Summary Table

January - December 1992

|                                |                                     |
|--------------------------------|-------------------------------------|
| Analyte                        | 2,3,7,8-tetrachlorodibenzo-p-dioxin |
| True Concentration             | 30 pg/L                             |
| Number of Observations         | 26                                  |
| Between-run Standard Deviation | 16 %                                |
| Accuracy (% of expected)       | 105 %                               |

## 1,2,3,7,8-pentachlorodibenzo-p-dioxin

recovery from effluent precision & recovery samples



..... average recovery ( % of expected )  
\_\_\_\_\_ 99% confidence limits  
- - - - - control limits

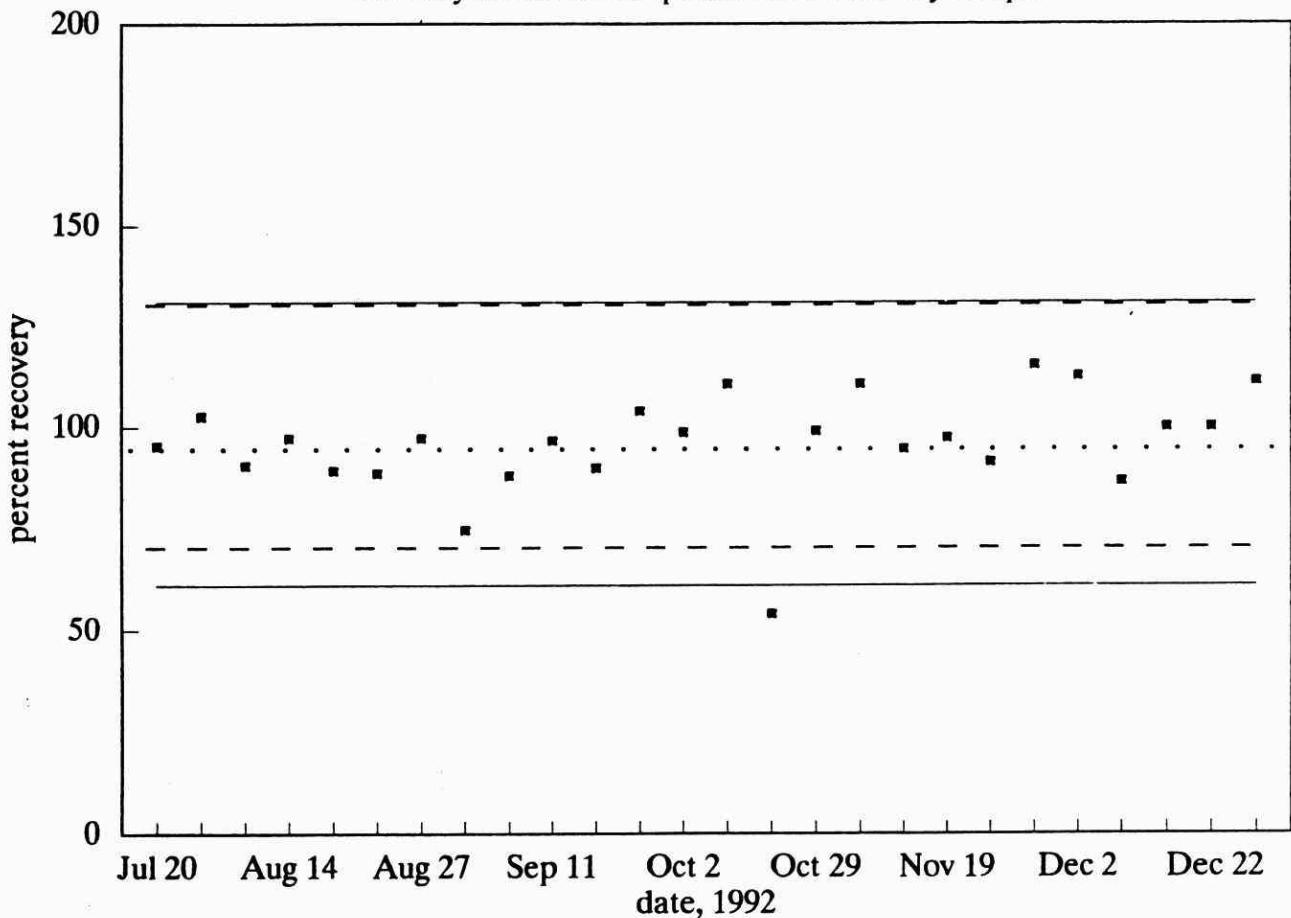
Performance Summary Table

January - December 1992

|                                |                                       |
|--------------------------------|---------------------------------------|
| Analyte                        | 1,2,3,7,8-pentachlorodibenzo-p-dioxin |
| True Concentration             | 150 pg/L                              |
| Number of Observations         | 26                                    |
| Between-run Standard Deviation | 20 %                                  |
| Accuracy (% of expected)       | 103 %                                 |

## 1,2,3,4,7,8-hexachlorodibenzo-p-dioxin

recovery from effluent precision & recovery samples



..... average recovery ( % of expected )  
\_\_\_\_\_ 99% confidence limits  
- - - - - control limits

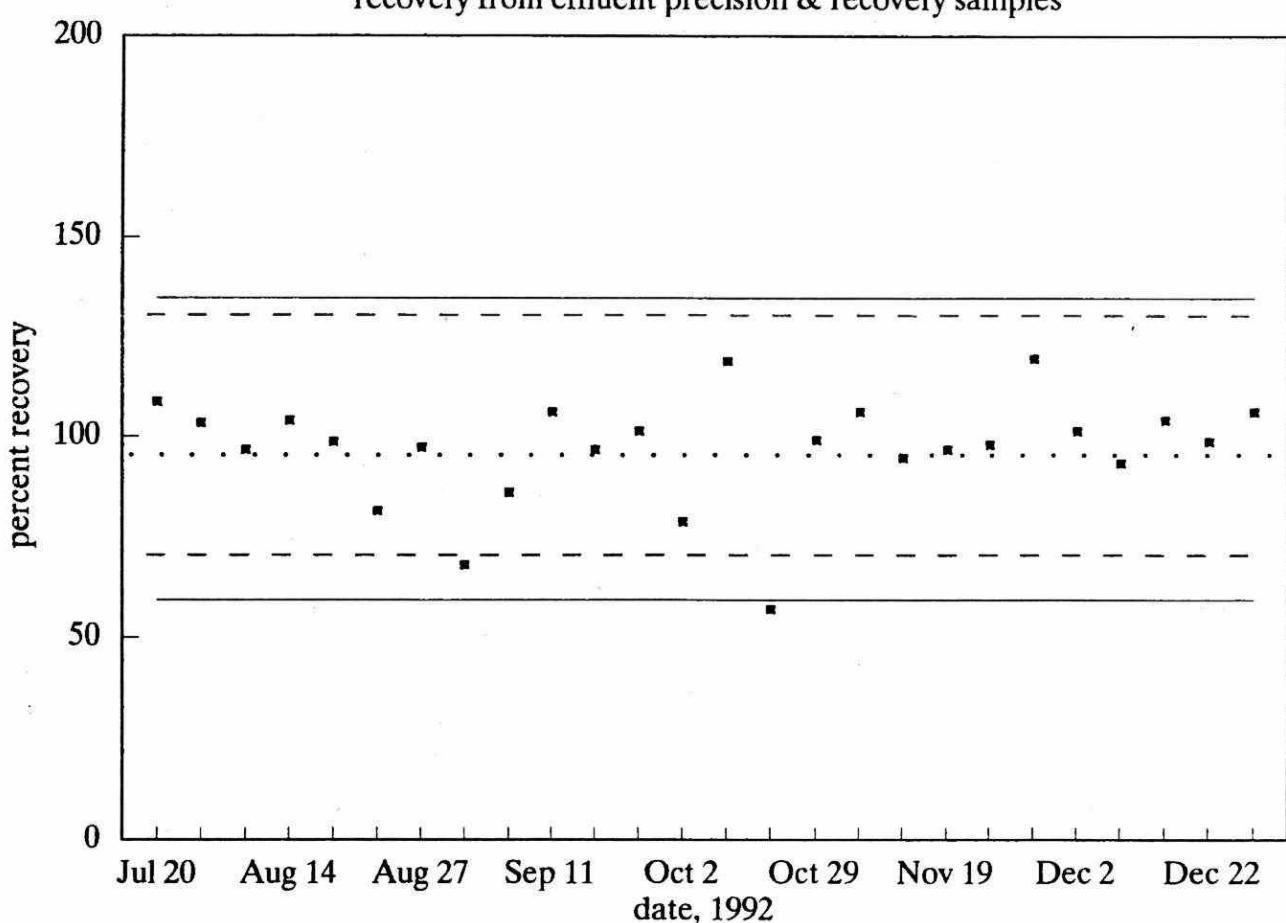
Performance Summary Table

January - December 1992

|                                |  |
|--------------------------------|--|
| Analyte                        | 1,2,3,4,7,8-hexachlorodibenzo-p-dioxin |
| True Concentration             | 150 pg/L                               |
| Number of Observations         | 26                                     |
| Between-run Standard Deviation | 13 %                                   |
| Accuracy (% of expected)       | 96 %                                   |

## 1,2,3,6,7,8-hexachlorodibenzo-p-dioxin

recovery from effluent precision & recovery samples



..... average recovery ( % of expected )  
\_\_\_\_\_ 99% confidence limits  
- - - - - control limits

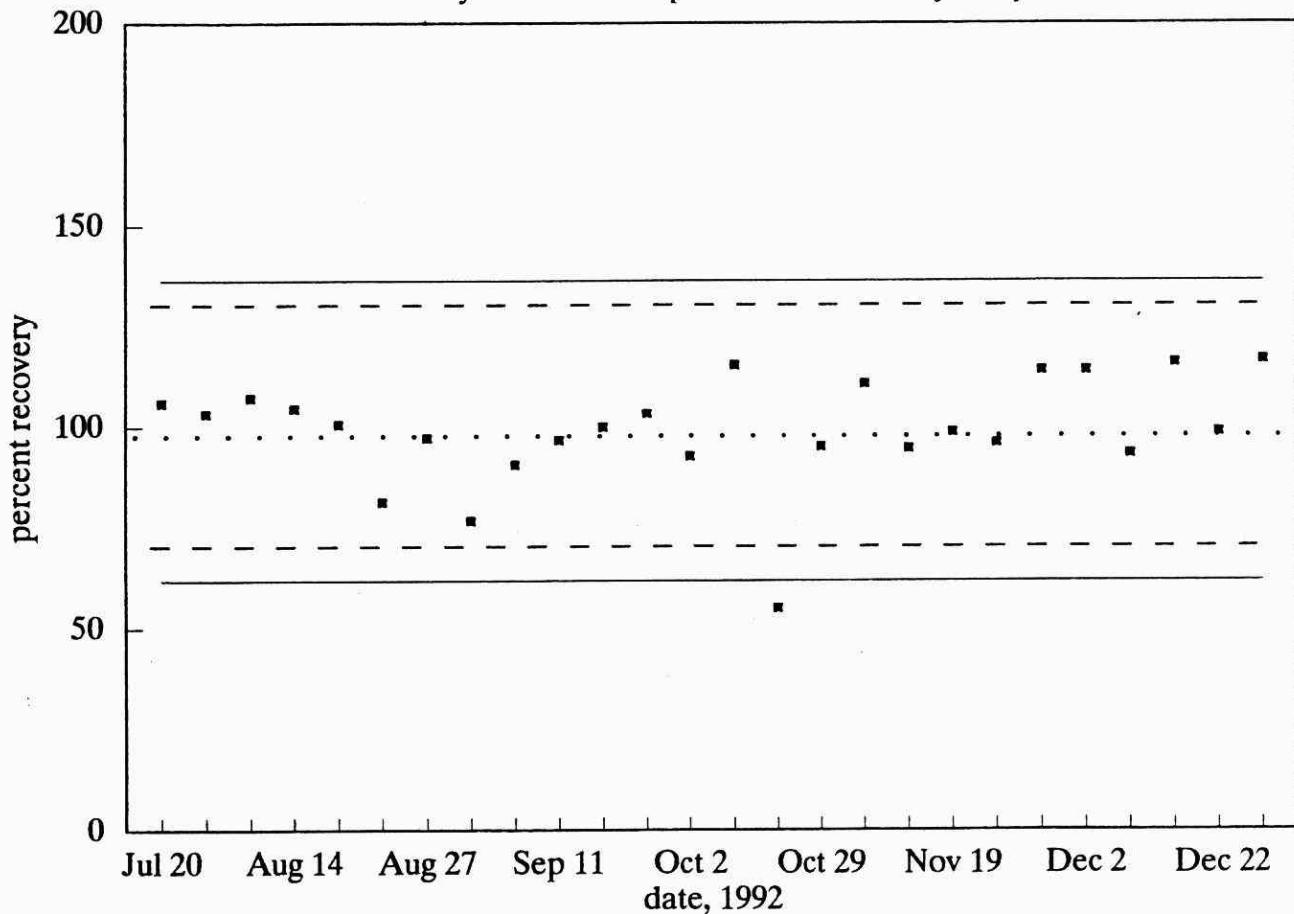
Performance Summary Table

January - December 1992

|                                |  |
|--------------------------------|--|
| Analyte                        | 1,2,3,6,7,8-hexachlorodibenzo-p-dioxin |
| True Concentration             | 150 pg/L                               |
| Number of Observations         | 26                                     |
| Between-run Standard Deviation | 14 %                                   |
| Accuracy (% of expected)       | 97 %                                   |

## 1,2,3,7,8,9-hexachlorodibenzo-p-dioxin

recovery from effluent precision & recovery samples



..... average recovery ( % of expected )  
\_\_\_\_ 99% confidence limits  
- - - - control limits

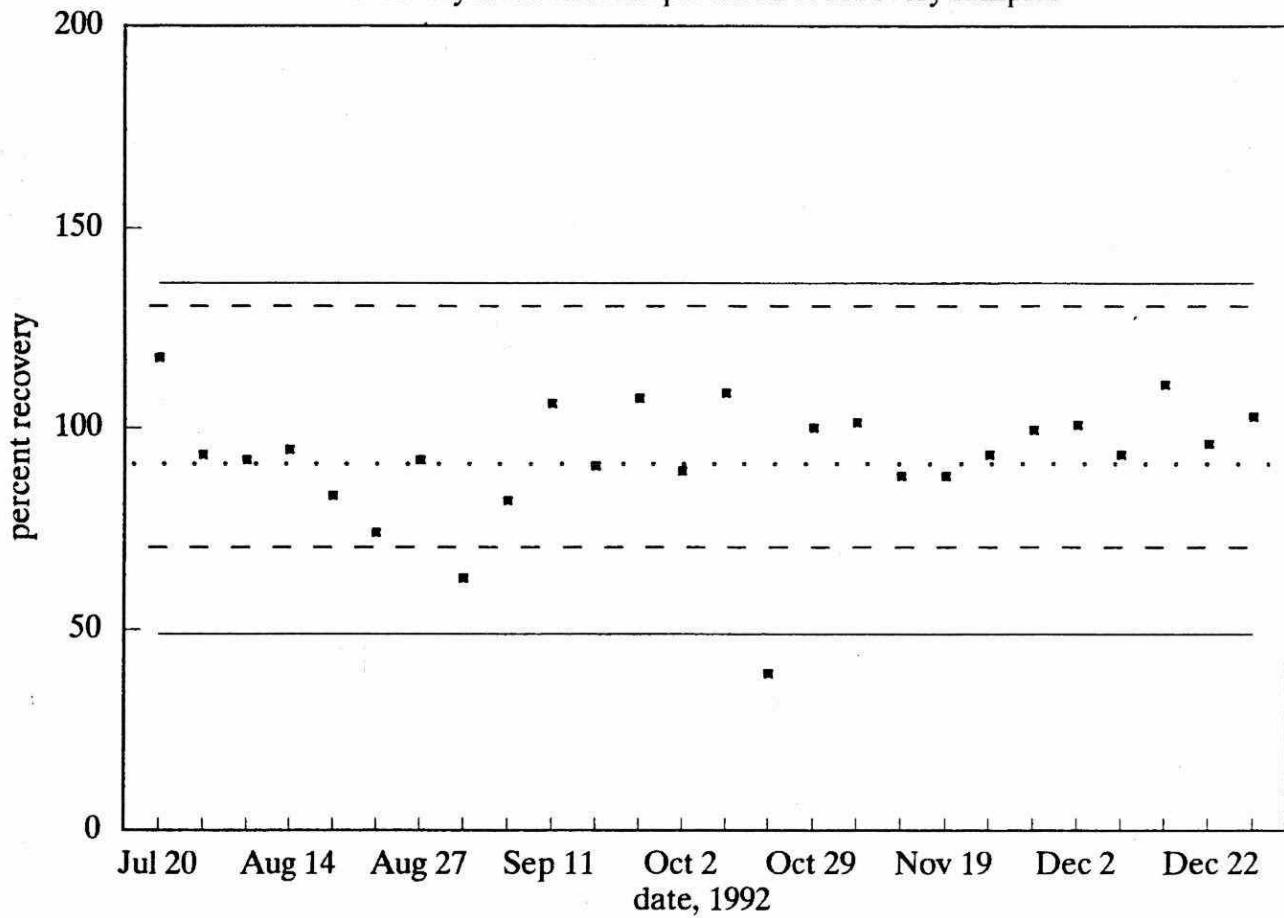
Performance Summary Table

January - December 1992

|                                |  |
|--------------------------------|--|
| Analyte                        | 1,2,3,7,8,9-hexachlorodibenzo-p-dioxin |
| True Concentration             | 150 pg/L                               |
| Number of Observations         | 26                                     |
| Between-run Standard Deviation | 14 %                                   |
| Accuracy (% of expected)       | 99 %                                   |

## 1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin

recovery from effluent precision & recovery samples



..... average recovery ( % of expected )  
\_\_\_\_\_ 99% confidence limits  
- - - - - control limits

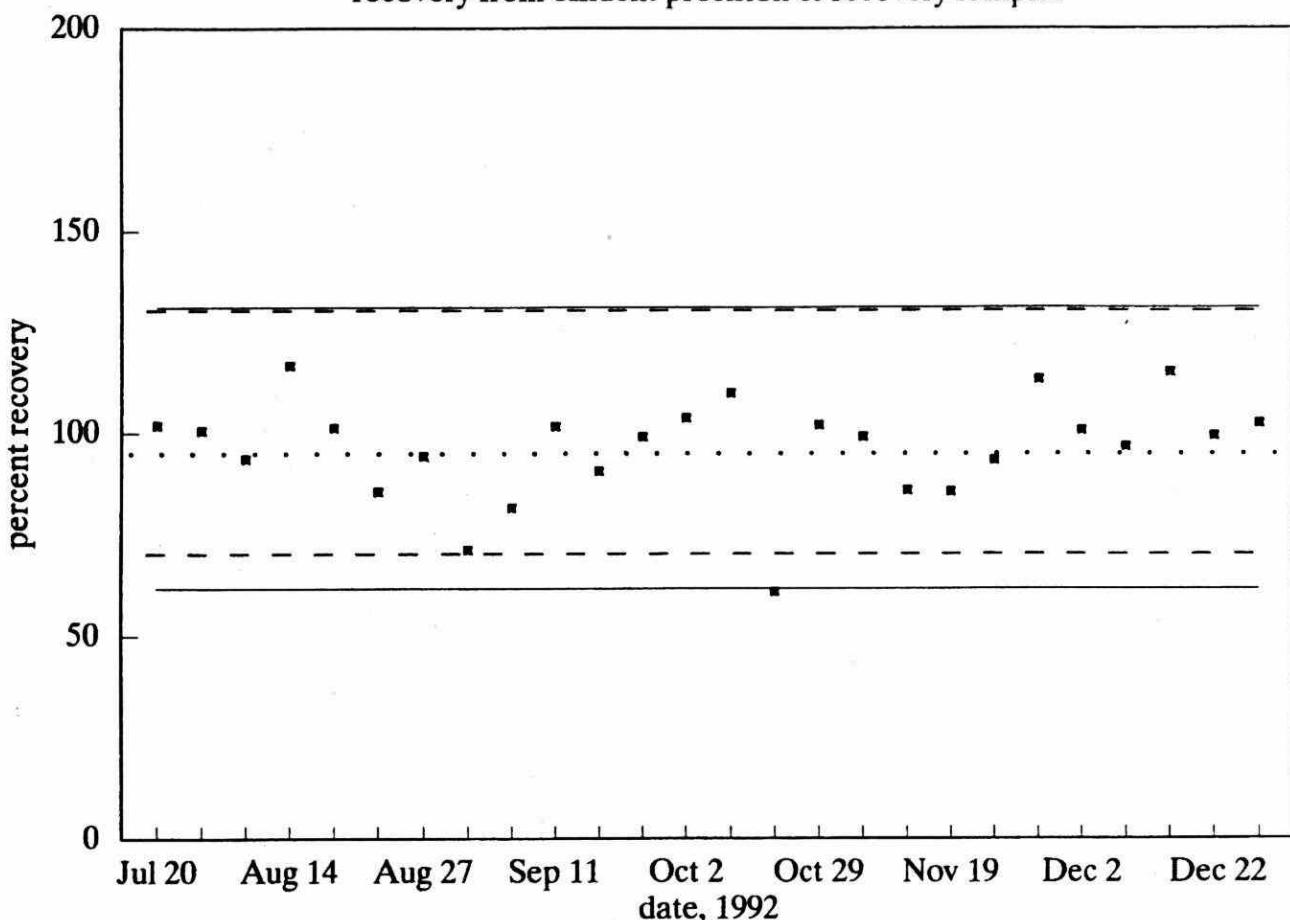
Performance Summary Table

January - December 1992

|                                |   |
|--------------------------------|---|
| Analyte                        | 1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin |
| True Concentration             | 150 pg/L                                  |
| Number of Observations         | 26  |
| Between-run Standard Deviation | 16 %                                      |
| Accuracy (% of expected)       | 93 %                                      |

## octachlorodibenzo-p-dioxin

recovery from effluent precision & recovery samples



..... average recovery ( % of expected )  
\_\_\_\_ 99% confidence limits  
- - - - control limits

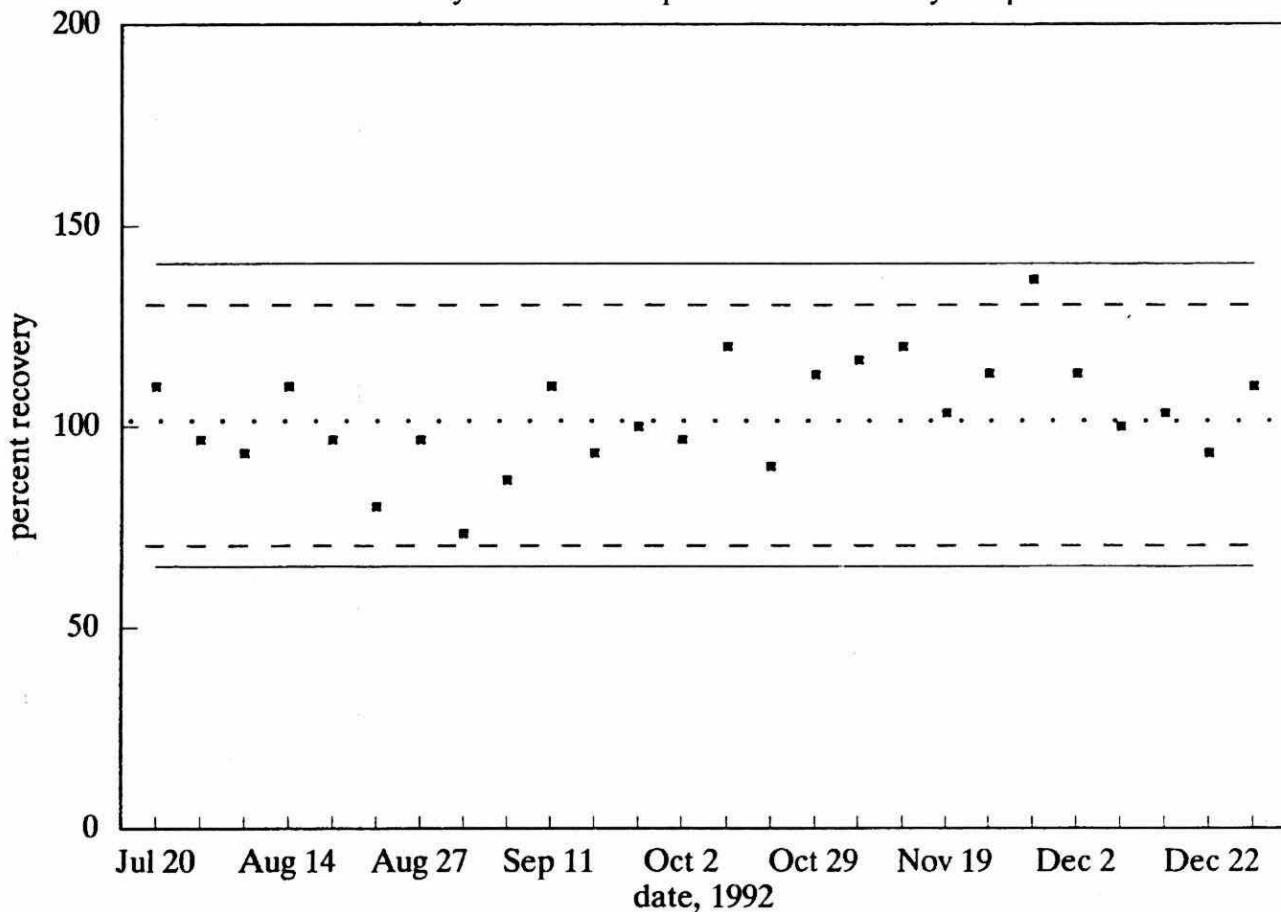
Performance Summary Table

January - December 1992

| Analyte                        | octachlorodibenzo-p-dioxin |
|--------------------------------|----------------------------|
| True Concentration             | 300 pg/L                   |
| Number of Observations         | 26                         |
| Between-run Standard Deviation | 13 %                       |
| Accuracy (% of expected)       | 96 %                       |

## 2,3,7,8-tetrachlorodibenzofuran

recovery from effluent precision & recovery samples



..... average recovery ( % of expected )  
\_\_\_\_\_ 99% confidence limits  
- - - - control limits

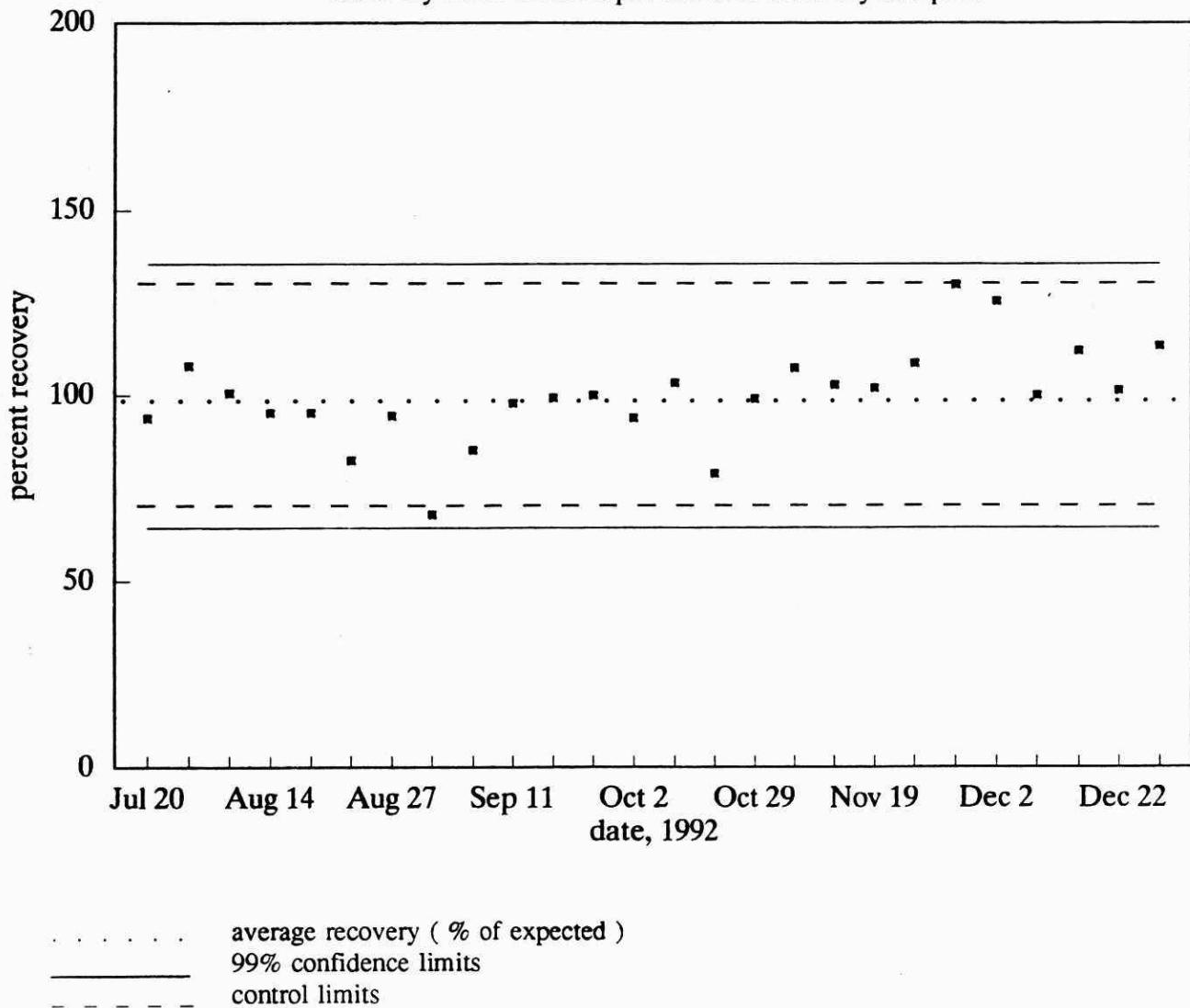
Performance Summary Table

January - December 1992

|                                |                                 |
|--------------------------------|---------------------------------|
| Analyte                        | 2,3,7,8-tetrachlorodibenzofuran |
| True Concentration             | 30 pg/L                         |
| Number of Observations         | 26                              |
| Between-run Standard Deviation | 14 %                            |
| Accuracy (% of expected)       | 103 %                           |

## 2,3,4,7,8-pentachlorodibenzofuran

recovery from effluent precision & recovery samples



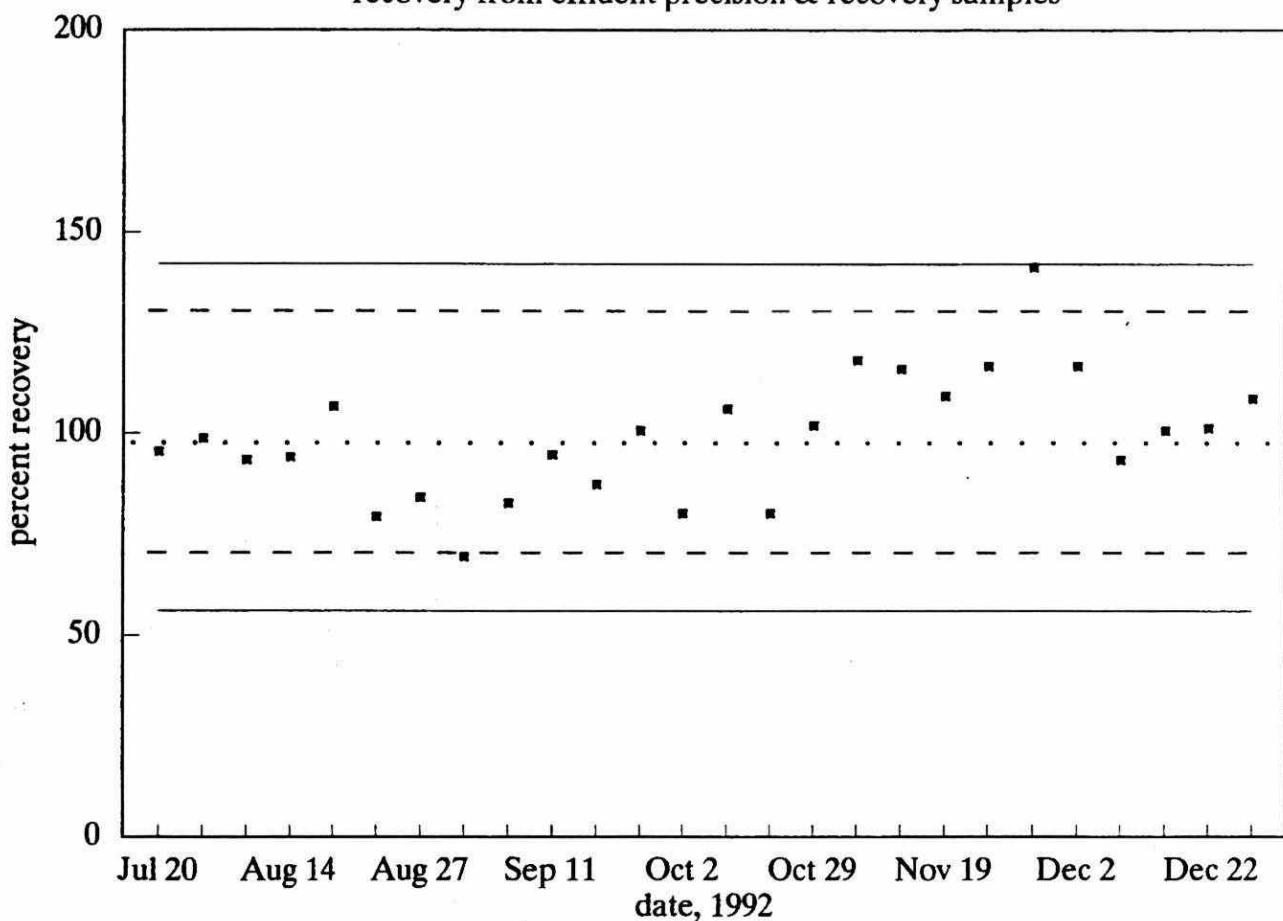
Performance Summary Table

January - December 1992

|                                |                                   |
|--------------------------------|-----------------------------------|
| Analyte                        | 2,3,4,7,8-pentachlorodibenzofuran |
| True Concentration             | 150 pg/L                          |
| Number of Observations         | 26                                |
| Between-run Standard Deviation | 13 %                              |
| Accuracy (% of expected)       | 100 %                             |

## 1,2,3,7,8-pentachlorodibenzofuran

recovery from effluent precision & recovery samples



..... average recovery ( % of expected )  
\_\_\_\_\_ 99% confidence limits  
- - - - - control limits

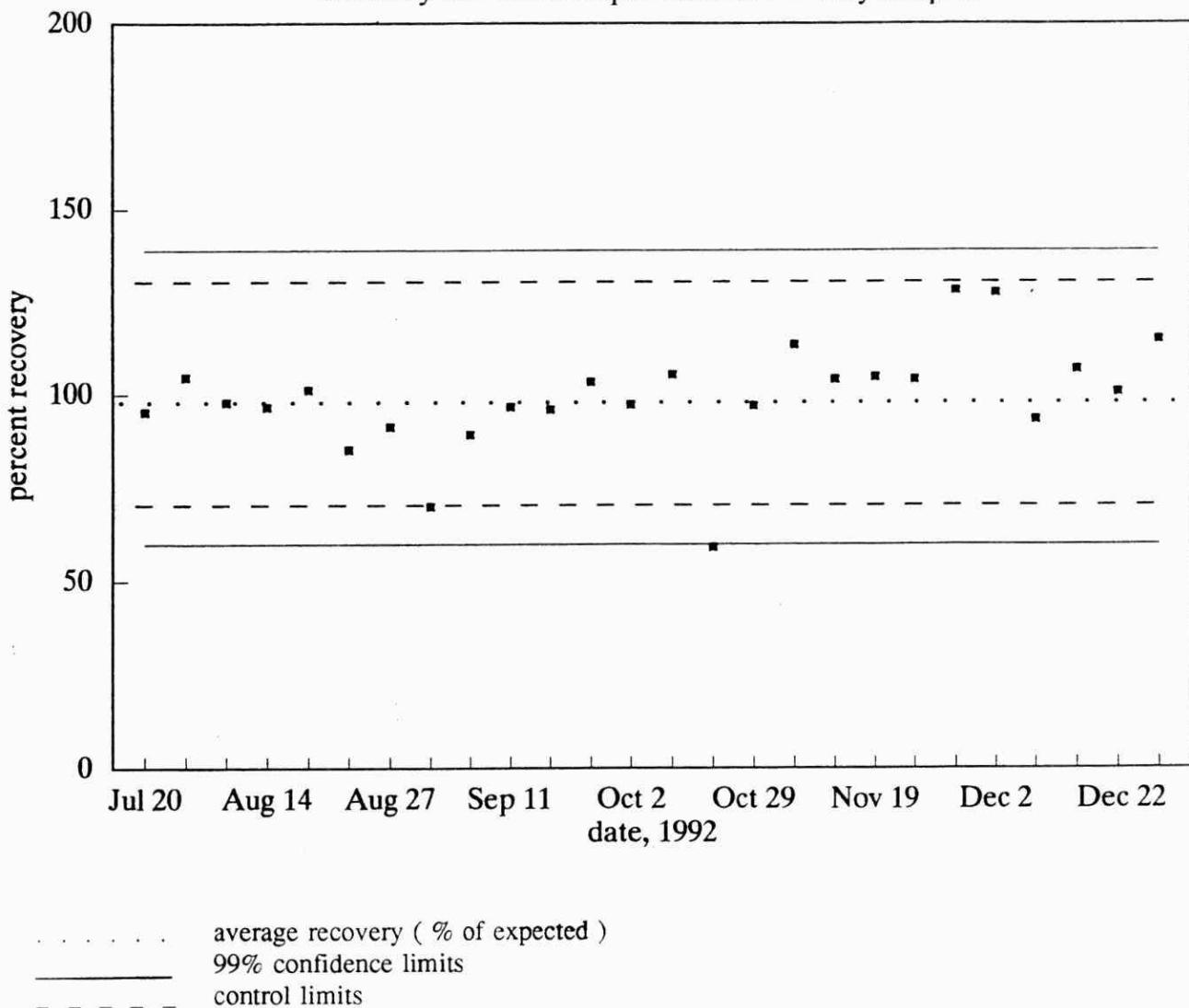
Performance Summary Table

January - December 1992

|                                |                                   |
|--------------------------------|-----------------------------------|
| Analyte                        | 1,2,3,7,8-pentachlorodibenzofuran |
| True Concentration             | 150 pg/L                          |
| Number of Observations         | 26                                |
| Between-run Standard Deviation | 16 %                              |
| Accuracy (% of expected)       | 99 %                              |

## 1,2,3,4,7,8-hexachlorodibenzofuran

recovery from effluent precision & recovery samples



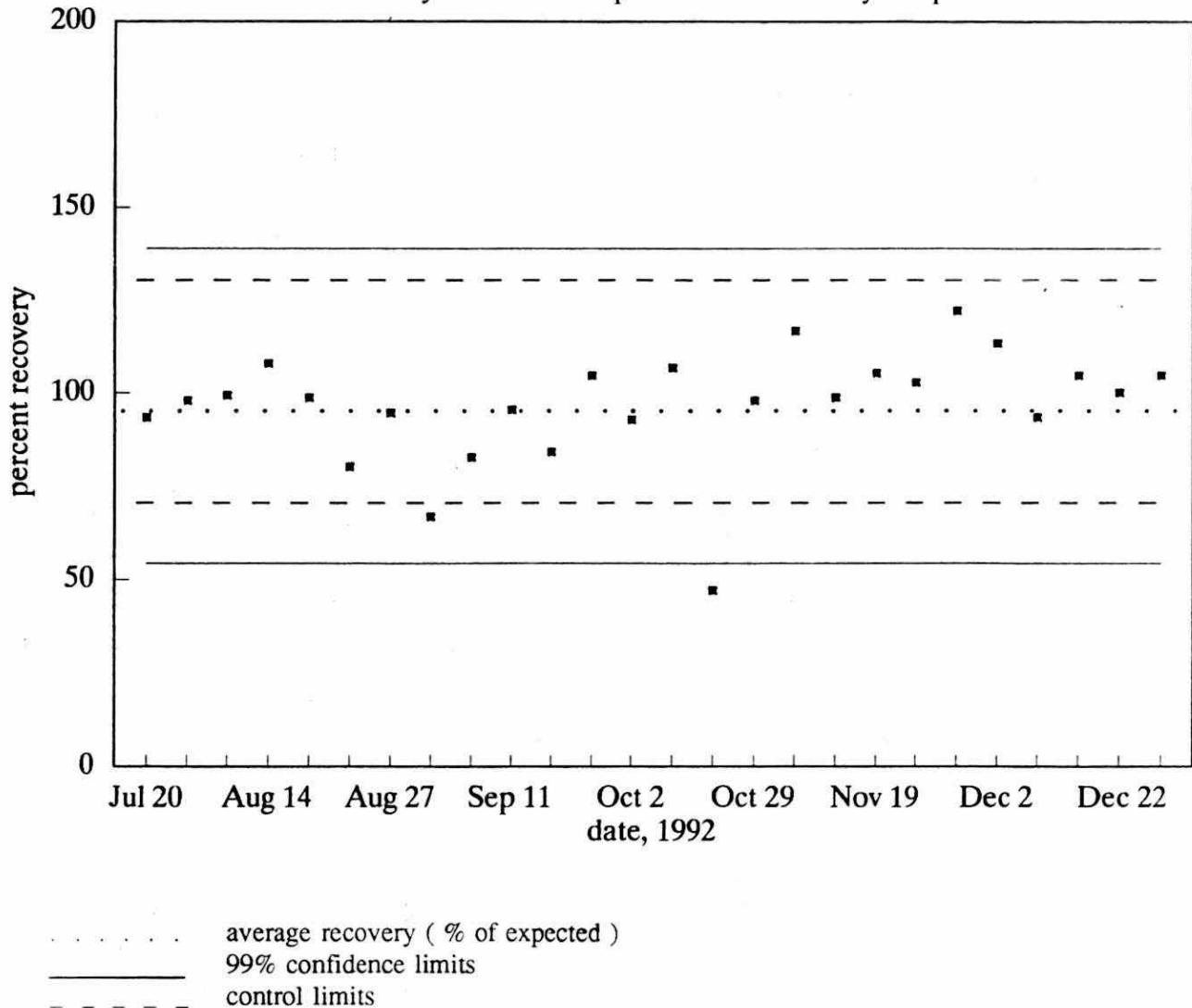
Performance Summary Table

January - December 1992

|                                |                                    |
|--------------------------------|------------------------------------|
| Analyte                        | 1,2,3,4,7,8-hexachlorodibenzofuran |
| True Concentration             | 150 pg/L                           |
| Number of Observations         | 26                                 |
| Between-run Standard Deviation | 14 %                               |
| Accuracy (% of expected)       | 99 %                               |

## 1,2,3,6,7,8-hexachlorodibenzofuran

recovery from effluent precision & recovery samples



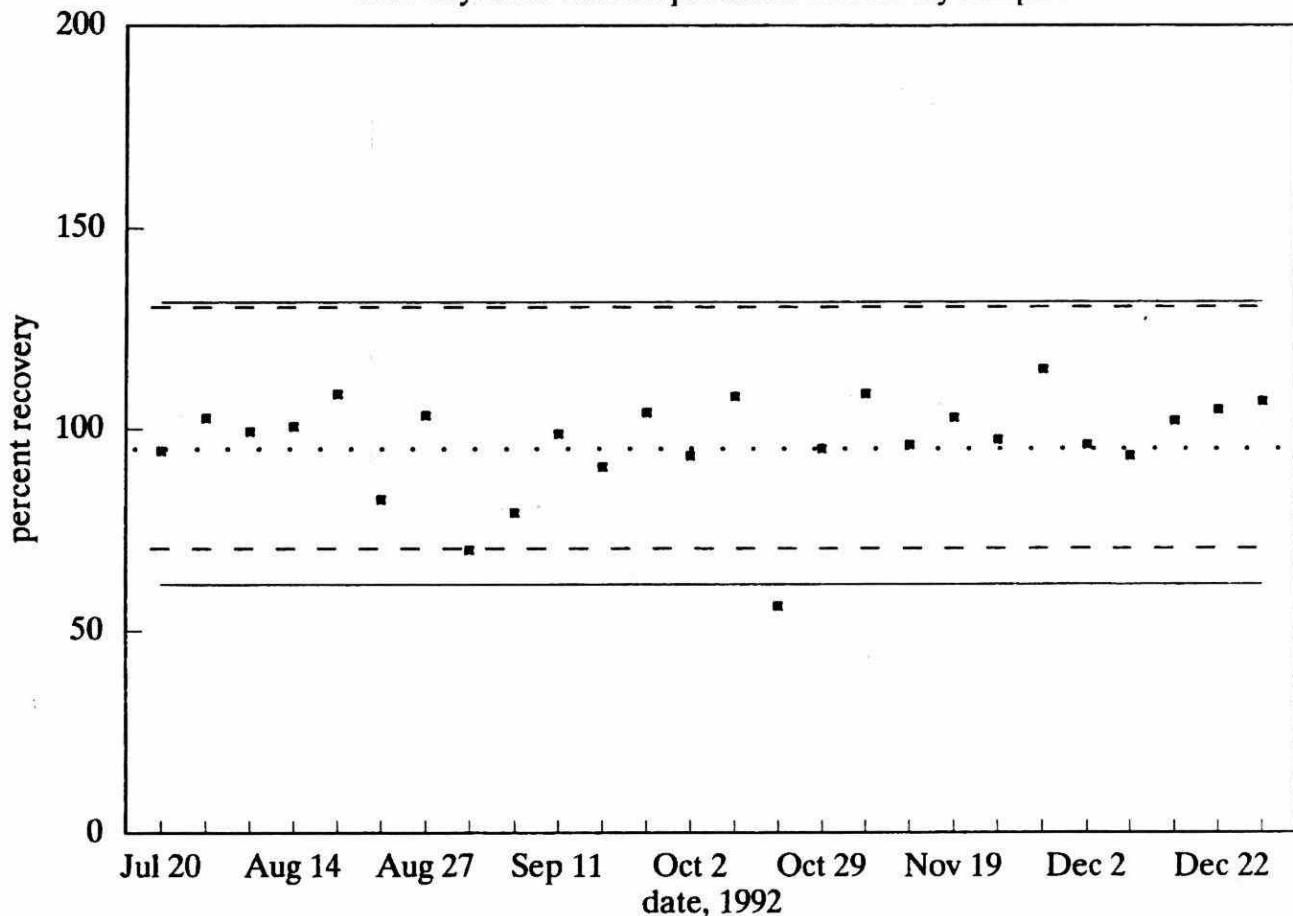
Performance Summary Table

January - December 1992

|                                |                                    |
|--------------------------------|------------------------------------|
| Analyte                        | 1,2,3,6,7,8-hexachlorodibenzofuran |
| True Concentration             | 150 pg/L                           |
| Number of Observations         | 26                                 |
| Between-run Standard Deviation | 15 %                               |
| Accuracy (% of expected)       | 97 %                               |

## 2,3,4,6,7,8-hexachlorodibenzofuran

recovery from effluent precision & recovery samples



..... average recovery ( % of expected )  
\_\_\_\_\_ 99% confidence limits  
- - - - - control limits

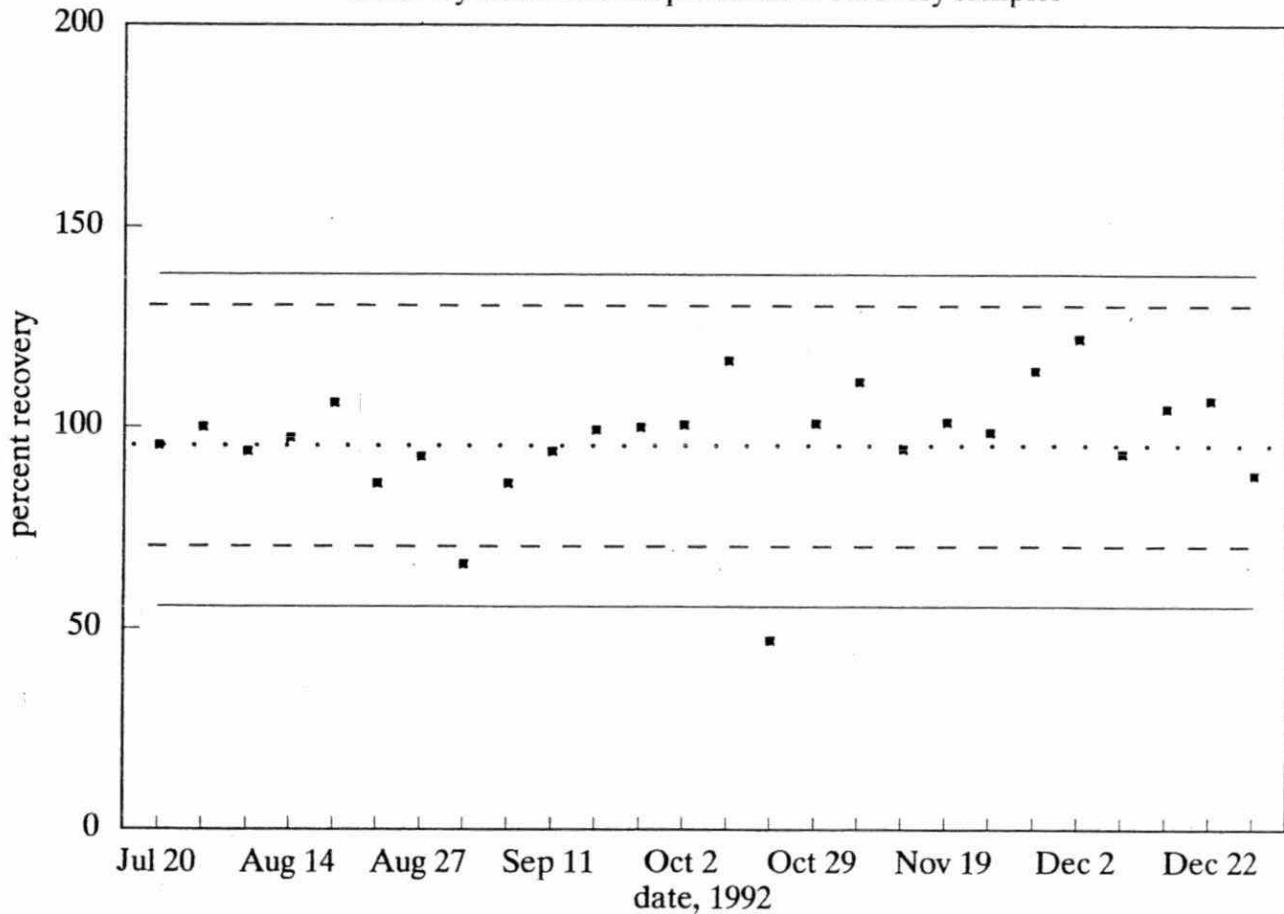
Performance Summary Table

January - December 1992

|                                |                                    |
|--------------------------------|------------------------------------|
| Analyte                        | 2,3,4,6,7,8-hexachlorodibenzofuran |
| True Concentration             | 150 pg/L                           |
| Number of Observations         | 26                                 |
| Between-run Standard Deviation | 13 %                               |
| Accuracy (% of expected)       | 97 %                               |

## 1,2,3,7,8,9-hexachlorodibenzofuran

recovery from effluent precision & recovery samples



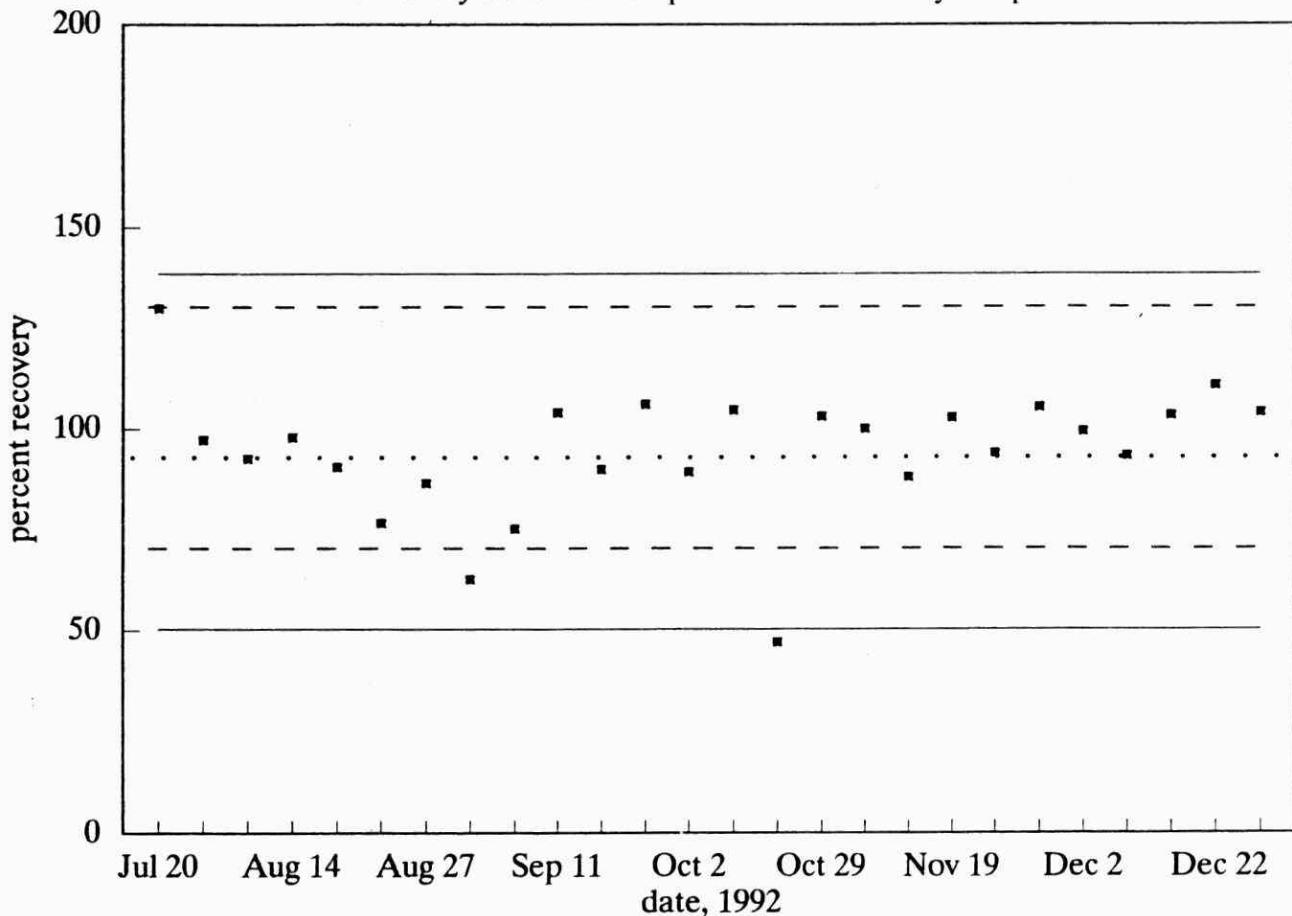
Performance Summary Table

January - December 1992

|                                |                                    |
|--------------------------------|------------------------------------|
| Analyte                        | 1,2,3,7,8,9-hexachlorodibenzofuran |
| True Concentration             | 150 pg/L                           |
| Number of Observations         | 26                                 |
| Between-run Standard Deviation | 15 %                               |
| Accuracy (% of expected)       | 97 %                               |

## 1,2,3,4,6,7,8-heptachlorodibenzofuran

recovery from effluent precision & recovery samples



..... average recovery ( % of expected )  
\_\_\_\_\_ 99% confidence limits  
- - - - - control limits

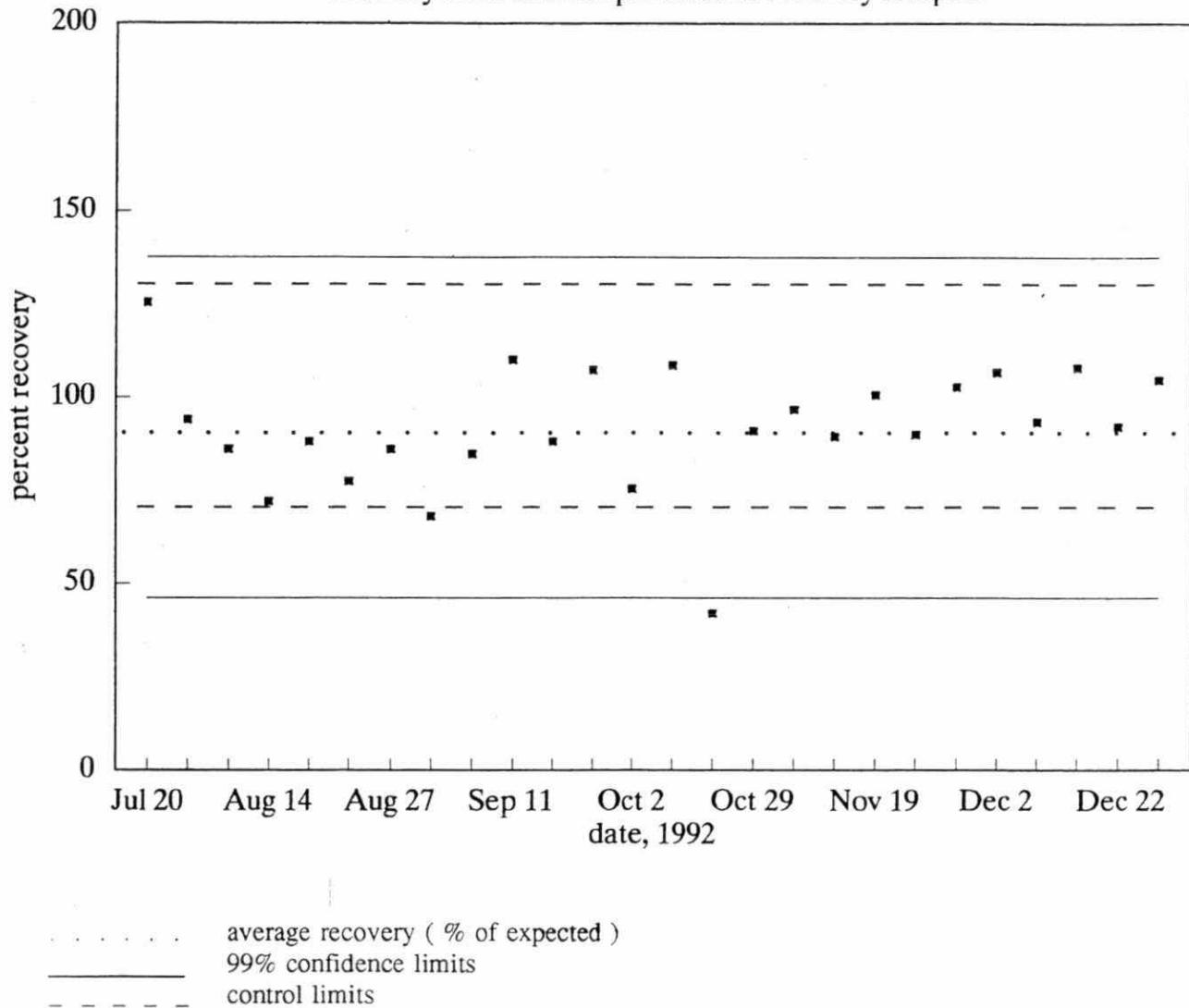
Performance Summary Table

January - December 1992

|                                |                                       |
|--------------------------------|---------------------------------------|
| Analyte                        | 1,2,3,4,6,7,8-heptachlorodibenzofuran |
| True Concentration             | 150 pg/L                              |
| Number of Observations         | 26                                    |
| Between-run Standard Deviation | 16 %                                  |
| Accuracy (% of expected)       | 94 %                                  |

## 1,2,3,4,7,8,9-heptachlorodibenzofuran

recovery from effluent precision & recovery samples



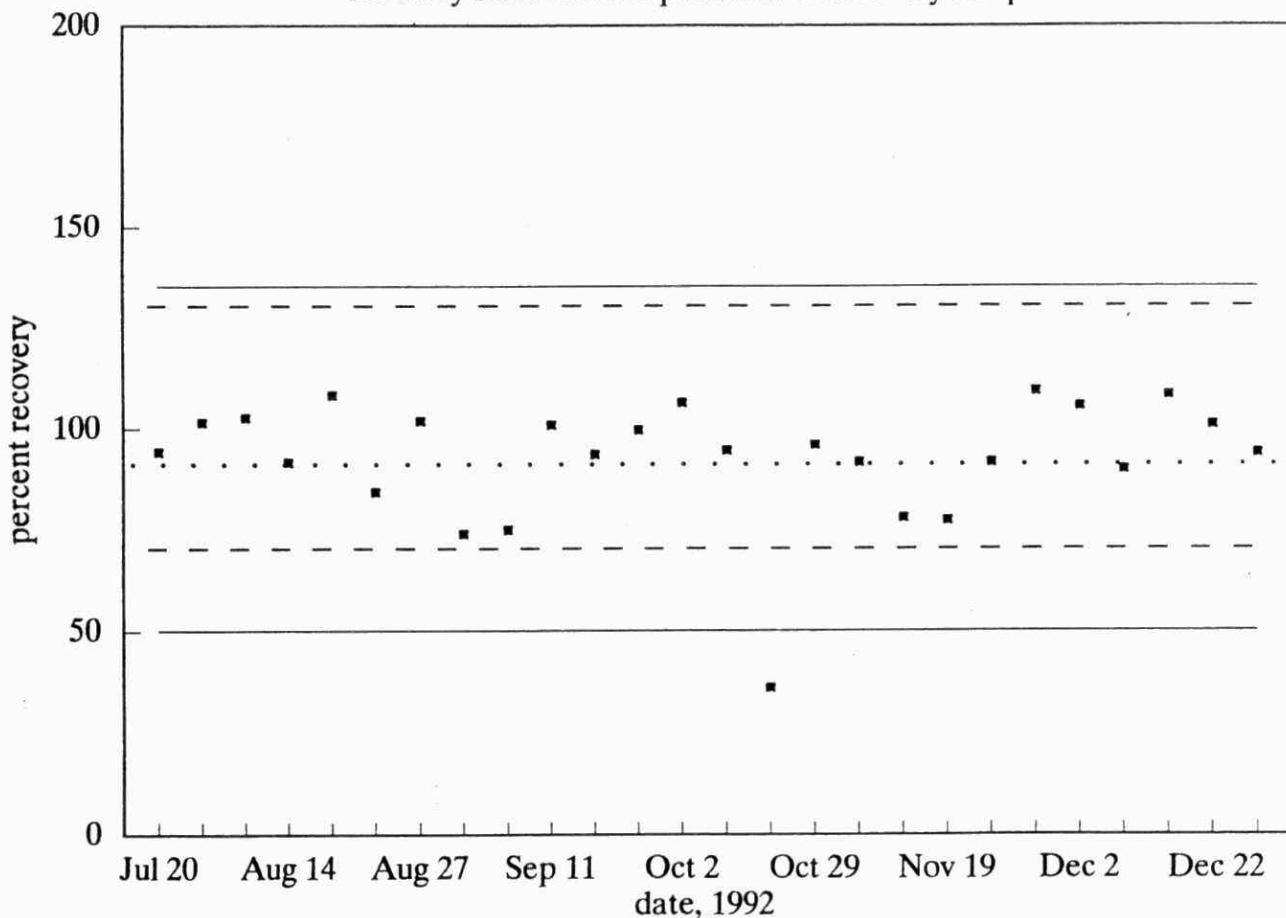
Performance Summary Table

January - December 1992

|                                |                                       |
|--------------------------------|---------------------------------------|
| Analyte                        | 1,2,3,4,7,8,9-heptachlorodibenzofuran |
| True Concentration             | 150 pg/L                              |
| Number of Observations         | 26                                    |
| Between-run Standard Deviation | 17 %                                  |
| Accuracy (% of expected)       | 92 %                                  |

## Octachlorodibenzofuran

recovery from effluent precision & recovery samples



..... average recovery ( % of expected )  
\_\_\_\_\_ 99% confidence limits  
- - - - - control limits

Performance Summary Table

January - December 1992

| Analyte                        | octachlorodibenzofuran |
|--------------------------------|------------------------|
| True Concentration             | 300 pg/L               |
| Number of Observations         | 26                     |
| Between-run Standard Deviation | 16 %                   |
| Accuracy (% of expected)       | 93 %                   |

**METHOD CODE :** PVAFD-E3317A

**METHOD TITLE:** The Determination of Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans in Vegetation by GC-MS

**LABORATORY :** Dioxin Unit

**SUPERVISOR :** Dr. E. Reiner

**SAMPLE TYPE :** vegetation

**PRINCIPLE OF THE METHOD :**

A known quantity of isotopically labelled PCDDs and PCDFs is added to each sample to serve as an internal standard. PCDDs and PCDFs are extracted from the sample using a Soxhlet extraction apparatus and a hexane/acetone mixture. A multi-stage chromatographic cleanup procedure is used to remove potential chemical interferences.

The reconstituted final extract is examined by gas chromatography - high resolution mass spectrometry (GC-HRMS) or gas chromatography/tandem mass spectrometry (GC-MS-MS).

**PARAMETERS MEASURED :**

**IDL ( pg/g )**

|   |   |
|---|---|
| 2,3,7,8-tetrachlorodibenzo-p-dioxin       | 1 |
| 1,2,3,7,8-pentachlorodibenzo-p-dioxin     | 2 |
| 1,2,3,4,7,8-hexachlorodibenzo-p-dioxin    | 3 |
| 1,2,3,6,7,8-hexachlorodibenzo-p-dioxin    | 3 |
| 1,2,3,7,8,9-hexachlorodibenzo-p-dioxin    | 3 |
| 1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin | 5 |
| octachlorodibenzo-p-dioxin                | 7 |
| 2,3,7,8-tetrachlorodibenzofuran           | 1 |
| 2,3,4,7,8-pentachlorodibenzofuran         | 2 |
| 1,2,3,7,8-pentachlorodibenzofuran         | 2 |
| 1,2,3,4,7,8-hexachlorodibenzofuran        | 3 |
| 1,2,3,6,7,8-hexachlorodibenzofuran        | 3 |
| 2,3,4,6,7,8-hexachlorodibenzofuran        | 3 |
| 1,2,3,7,8,9-hexachlorodibenzofuran        | 3 |
| 1,2,3,4,6,7,8-heptachlorodibenzofuran     | 5 |
| 1,2,3,4,7,8,9-heptachlorodibenzofuran     | 5 |
| octachlorodibenzofuran                    | 7 |

total tetrachlorinated dibenzo-p-dioxins ( TCDD )  
total pentachlorinated dibenzo-p-dioxins ( PCDD )  
total hexachlorinated dibenzo-p-dioxins ( HxCDD )  
total heptachlorinated dibenzo-p-dioxins ( HpCDD )  
total tetrachlorinated dibenzofurans ( TCDF )  
total pentachlorinated dibenzofurans ( PCDF )  
total hexachlorinated dibenzofurans ( HxCDF )  
total heptachlorinated dibenzofurans ( HpCDF )

### REPORTING FORMAT :

Results are reported in parts per trillion ( pg/g ) rounded off to 2 significant figures. The minimum reported levels are sample and analyte specific \* and range from 1 ppt to 10 ppt.

### QUALITY CONTROL :

The routine quality control operations monitor overall method performance ( precision and recovery samples ), validity of calibration and consistency in injection volume ( injection standard ), absence of potential contamination ( method blanks ) and recovery of target analytes ( internal quantitation standard ).

#### List of Performance Tables : Method Blanks Summary

| Method Blanks Summary                     |                        | January 1992 - December 1992   |                             |  |
|---|------------------------|--------------------------------|-----------------------------|--|
| Analyte                                   | Number of Observations | Average Concentration ( pg/g ) | Standard Deviation ( pg/g ) |  |
| 2,3,7,8-tetrachlorodibenzo-p-dioxin       | 4                      | ND ( 1 )                       |                             |  |
| 1,2,3,7,8-pentachlorodibenzo-p-dioxin     | 4                      | ND ( 2 )                       |                             |  |
| 1,2,3,4,7,8-hexachlorodibenzo-p-dioxin    | 4                      | ND ( 3 )                       |                             |  |
| 1,2,3,6,7,8-hexachlorodibenzo-p-dioxin    | 4                      | ND ( 3 )                       |                             |  |
| 1,2,3,7,8,9-hexachlorodibenzo-p-dioxin    | 4                      | ND ( 3 )                       |                             |  |
| 1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin | 4                      | ND ( 5 )                       |                             |  |
| octachlorodibenzo-p-dioxin                | 4                      | 9.0                            | 9.0                         |  |
| 2,3,7,8-tetrachlorodibenzofuran           | 4                      | ND ( 1 )                       |                             |  |
| 2,3,4,7,8-pentachlorodibenzofuran         | 4                      | ND ( 2 )                       |                             |  |
| 1,2,3,7,8-pentachlorodibenzofuran         | 4                      | ND ( 2 )                       |                             |  |
| 1,2,3,4,7,8-hexachlorodibenzofuran        | 4                      | ND ( 3 )                       |                             |  |
| 1,2,3,6,7,8-hexachlorodibenzofuran        | 4                      | ND ( 3 )                       |                             |  |
| 2,3,4,6,7,8-hexachlorodibenzofuran        | 4                      | ND ( 3 )                       |                             |  |
| 1,2,3,7,8,9-hexachlorodibenzofuran        | 4                      | ND ( 3 )                       |                             |  |
| 1,2,3,4,6,7,8-heptachlorodibenzofuran     | 4                      | ND ( 5 )                       |                             |  |
| 1,2,3,4,7,8,9-heptachlorodibenzofuran     | 4                      | ND ( 5 )                       |                             |  |
| octachlorodibenzofuran                    | 4                      | 0.9                            | 1.5                         |  |

ND ... Not detected. Detection limits in pg/g given in brackets ( ).

\* The minimum reported levels correspond to the amount of analyte that would give most-abundant ion response five times higher than corresponding instrumental noise.

**QD  
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1992**

1992 performance report :  
drinking water organics section  
/ Duchoslav, Eva (ed.)  
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